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Research Materials Developed Under the NBS Inorganic Materials Program

Edited by
F. E. Brinckman and J. B. Wachtman, Jr.

Inorganic Materials Division
Institute for Materials Research
National Bureau of Standards
Washington, D.C. 20234



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Research Materials Developed Under the NBS Inorganic Materials Program

F. E. Brinckman and J. B. Wachtman, Jr.

The National Bureau of Standards develops many specialized materials in the process of carrying out research supporting its measurements, standards, and service activities. These materials include gases, liquids, glasses, single crystals, polycrystals, and various multiphase materials; their compositions (including trace elements in some cases) and physical characteristics are tailored to specific research needs, but the materials are often of use for other research purposes. Production is usually limited to immediate internal needs and samples are not generally available for distribution, but knowledge of production techniques and materials characteristics which may be helpful to other scientists is available. The present listing of research materials developed in the Inorganic Materials Division accordingly gives names of scientific staff members who may be contacted for this type of information as well as giving a brief summary of the nature, method of preparation and properties determined for each material.

Key words: Chemical properties; composition; gases; glasses; liquids; multiphase materials; physical properties; polycrystals; preparation; research.

1. Introduction

Improved materials are critically needed in many areas of technology. Attempts to provide such superior materials are frequently empirical and sometimes wasteful of time and funds [1].¹ An empirical component will probably always be present in any materials development program, but the establishment of a science of materials that increasingly affords predictable and reliable results in devising new materials for specific tasks is most desirable. Not only will this increase the efficiency of materials development programs, but also can ultimately lead to development of completely new materials or new applications. Much thought has been given to opportunities arising from recent developments in materials science [2] and to roadblocks preventing engineering achievement of scientifically feasible materials including the areas of characterization [1] and processing [3]. Advances in materials science assist materials development on the one hand, but depend upon development of materials suitable for research on the other. This interdependence of materials development and materials science is widely recognized and new programs on the properties of materials increasingly have as a major component a research material development subprogram. The experimental and theoretical competence needed to produce and characterize specimens is frequently quite different from that needed for the subsequent property study so that the decision to undertake a property study on a new material frequently means that a large investment of time and resources, perhaps comparable to the property measuring effort itself, must be made before the property study can begin. In these circumstances the availability of a few trial specimens, even if not of completely satisfactory quality for the final study, can be very valuable in permitting a test of the experimental feasibility of the contemplated measurements and perhaps in providing guidance on how closely the character of the specimen must be controlled for the final study. When trial specimens are not available, knowledge of previous preparation techniques and of the nature and quality of specimens which have

been produced successfully is usually quite valuable in planning and carrying out the sample preparation aspect of a new research program. The present listing of research materials produced in the Inorganic Materials Division is offered as a means of promoting direct scientist-to-scientist exchange of information on production and characterization of materials for which the division has some special competence. In some cases, specimens may be on hand and available on request; in other cases, facilities and staff may be available to produce additional specimens for purposes coming within the NBS mission. Provision of information, rather than specimens, is the principal goal, however, because maintenance of a stock of the numerous and extremely varied materials produced in small batches or maintenance of specialized production facilities on a standby basis is not feasible.

Information concerning nationwide sources of many research materials, especially single crystals, is available from the Oak Ridge Research Materials Information Center [4].

The present listing of materials may be useful to scientists and engineers interested in the production of a material or its general availability for purposes other than research. No claim is made concerning exhaustive knowledge of the materials listed, but the staff members involved in the production or characterization of unusual materials sometimes have special knowledge relevant to other applications in addition to research.

2. Definition of Research Materials

2.1. General Definition

A research material is here considered to be one sufficiently well-characterized to be useful for a particular type of current research. Ideally, a fully characterized material is desirable; that is, the character (chemical composition, structure, microstructure, etc.) should be uniform and held within such narrow and known limits that all the chemical and physical properties of the material are well determined. Practical considerations usually limit the characterization to

¹ Figures in brackets indicate the literature references on page 3.

the factors thought to control the property being studied and limit the accuracy of the characterization. Thus, a material useful at a particular stage of research may no longer be useful for the same type of research as measurement techniques improve and finer details of behavior are studied or as new factors are discovered to have an influence on properties at the level being studied. A valid research material can cease to have this distinction as a field of study progresses but the same material can sometimes again become an important research material as new phenomena are investigated. For example, ruby grown by the Verneuil process for bearings, wear surfaces, or jewels suddenly became an exciting research material when the ruby laser was invented.

2.2. Differences in Degree of Characterization Needed for Research on Properties with Varying Degrees of Structure Sensitivity.

The characterization needed for certain research, such as phase equilibria, is sometimes limited primarily to the major element composition and such factors as trace impurities or surface condition are relatively unimportant. This fact is reflected in the listing, for example, of a number of crystalline materials developed for phase equilibria studies and a number of glasses developed for bulk optical or elasticity studies; major component characterization was sufficient for these purposes. Some of these materials have subsequently become the object of renewed research interest in connection with other properties dependent primarily upon major component composition such as electro-optic properties, photoelasticity, and mechanical properties under pressure. Certain structure-sensitive properties, such as transport, plastic deformation, and fracture depend upon trace impurities and upon small deviations from stoichiometry. Samples made for research in these areas, even though of the same major component composition as some of those mentioned previously, require more careful processing and characterization and are accordingly listed as separate entries with an indication of their special features.

2.3. Inhomogeneity

Among both the bulk composition and trace impurity types there are cases where a deliberate degree of inhomogeneity is required. Thus, a series of glasses with the same total composition but differing degrees of phase separation forms an interesting family of research materials. An example of an even finer scale of deliberately produced inhomogeneity is a series of crystals of CaF_2 all having the same level of Gd additive but differing in the degree of association of point defects with Gd atoms. This last example illustrates another feature of some of the listed materials; in this case specimens of given bulk composition were produced by a commercial supplier to NBS specifications. The NBS contribution was the development of special treatments and measurement tech-

niques needed to produce and determine different degrees of association.

2.4. Metastable Materials

Still another category of research materials involves those which are not stable or have a very short lifetime under ordinary conditions but which occur as important components under special conditions such as high temperature vapor species, high pressure crystalline phases, or short-lived reactive intermediates. Such materials generally cannot be kept in stock but the technology of preparation and the techniques for measurement of concentration and properties during the brief lifetime of the material are important aspects of research material information. An overlapping family of research materials consists of those which are dangerous (toxic, explosive, or corrosive) and so require special handling procedures during processing, storage, or property measurement. An example of such a difficult research material is the high pressure polymorph of the detonator explosive lead azide.

3. Relation to Standard Reference Materials

A few of the research materials listed here have been developed into Standard Reference Materials and many of the Standard Reference Materials are used as research materials but the two categories should not be confused despite the fact that there is some overlap. Standard Reference Materials are kept in stock by the National Bureau of Standards and sold by the Office of Standard Reference Materials. Each Standard Reference Material is certified with respect to the aspects of chemical and/or physical properties relevant to the material's intended application. Perhaps the greatest use of these materials is for calibration of instruments and checking of measurement procedures, but the high degree of homogeneity and extensive characterization associated with Standard Reference Materials frequently makes them useful as research materials. A full listing of Standard Reference Materials is available [5].

4. Organization of the Tables

4.1. Rationale

As noted in the Introduction, entries have been tabulated into sections derived chiefly from gross physical state at ordinary conditions (e.g., gases, polycrystals, multiphases, etc.). Clearly a number of exceptions occur, particularly for research material of low stability or transient existence, but their location will be fairly obvious to the reader.

Since this compilation tends to emphasize composition rather than properties, special care was taken in organizing the tables. Use of the widely accepted Chemical Abstracts Formula Index does not always generate familiar empirical chemical formulae, no

does this system readily lend itself to classification of glasses, but we chose it for its rational basis. Moreover, significant progress has been achieved for computer searching and retrieval; indeed, the parent program is now in use by the Patent Office.

4.2. Use of Tables—Key

a. The arrangement of symbols in formulae is alphabetic except that in carbon compounds C always comes first followed immediately by H if hydrogen is also present.

b. The arrangement of formulae or entries is also alphabetic, except that the number of atoms of any specific kind influences the order of listing; e.g., all C_1 compounds appear before C_2 , thus CCl_2O , CCl_4 , $CHCl_3$, CHN , CH_2O , CO , C_2Ca , $C_2H_4O_2$.

c. Water of hydration is not made a part of the formulae indexed.

d. Polymers having different names and recognized as different substances, e.g., acetaldehyde and paraldehyde, are all entered under their accepted formulae; but a definite compound for which different polymeric formulae are in use is entered under the simplest formula.

e. For series of compounds listed under a single entry heading "M" refers to metallic or metalloidal components (e.g., K, Li, Rb) and "X" denotes electronegative substituents such as Cl, F, O, S, etc.

f. Approximate compositions are indicated by enclosing listing in quotation marks, "—", such as in interstitial compounds or alloys.

g. Glasses, polyphases, and compounds of given stoichiometry but indefinite structure are cited alphabetically by M, followed by X; thus $B_2O_3 \cdot 3Nb_2O_5$; $Cr_2O_3 \cdot IrO_2$; $B_2O_3 \cdot BaO \cdot SiO_2$; etc.

h. Isotopic compositions are presumed to be of terrestrial abundance unless otherwise indicated.

i. Wherever practicable conventional formulae and/or nomenclature is listed to the right of the index formula.

j. Great effort has been made to maintain accuracy and provide current information. In some instances, through staff changes, some authors cited are no

longer in the Inorganic Materials Division, yet inclusion of their recent efforts was deemed significant to this compendium. Hence, these individuals are signified by an asterisk. The reader may contact these individuals for further information, but it would probably be more expedient if the editors were contacted.

Many of the materials listed in this report were produced as part of programs sponsored by other agencies. Acknowledgements for specific materials are given in the references and are too numerous to repeat in detail here. The support by the Air Force Materials Laboratory, the Air Force Cambridge Research Laboratory, the Army Research Office (Durham), the Advanced Projects Research Agency (Materials Office), the Atomic Energy Commission (Research Division), the National Aeronautics and Space Administration, and the Office of Naval Research is gratefully acknowledged.

5. References

- [1] Characterization of Materials, prepared by the Committee on Characterization of Materials of the Materials Advisory Board, H. W. Leverenz, Committee Chairman. National Academy of Sciences—National Academy of Engineering Publication MAB-229-M, March 1967.
- [2] Research in Solid-State Sciences—Opportunities and Relevance to National Needs, prepared by a Committee of the Solid State Sciences Panel for the Coordinating Committee for Materials Research and Development of the Office of Science and Technology, R. Smoluchowski, Committee Chairman. Publication 1600, National Academy of Sciences, 1968.
- [3] Ceramic Processing, prepared by the Committee on Ceramic Processing, of the Materials Advisory Board, Joseph A. Pask, Committee Chairman. Publication 1576, National Academy of Sciences, 1968.
- [4] J. W. Cleland, Purity and Perfection of Research Specimens of Oxides in Mass Transport in Oxides, Nat. Bur. Stand. (U.S.), Spec. Pub. 296, (224 pages) edited by J. B. Wachtman, Jr. and A. D. Franklin, issued August 1968. The address of the Research Materials Information Center is Building 3001, Oak Ridge National Laboratory, Box X, Oak Ridge, Tennessee 37830.
- [5] Catalog of Standard Materials, Nat. Bur. Stand. (U.S.), Spec. Publ. 260, July 1970. See also supplements for price lists and changes.



I. GASES

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
$\text{BCl}_n\text{F}_{3-n}$	equilibrium soln. of all species, where $n = 0-3$, studied as liquids	$\text{BCl}_3 + \text{BF}_3 \rightleftharpoons$ at $< 25^\circ\text{C}$	NMR (^{11}B , ^{19}F) spectrometry	attempt to determine chemical exchange rates in equilibrium	T. C. Farrar	
BF_2H HBF_2 difluoroborane	also studied as liquid (1,2)	(3) $\text{BF}_3 + \text{B}_2\text{H}_6$ at 250°C (4) $\text{BF}_3 + (\text{CH}_3\text{O})_2\text{BH}$ $\text{B}_2\text{F}_4 + (\text{CH}_3)_3\text{SiH} + h\nu$	mol. wt., infrared NMR (^1H , ^{11}B , ^{19}F) spectrometry, active H, chemical reactivity	qualitative structural detms; only known monoboron fluoro-hydride; synth. reagent; air/moisture sensitive	T. D. Coyle T. C. Farrar	(1) T. C. Farrar and T. D. Coyle, "Proton and Fluorine NMR Spectra of HBF_2 ", J. Chem. Phys., <u>41</u> , 2612 (1964). (2) E. B. Whipple, T. H. Brown, T. C. Farrar, and T. D. Coyle, "Relative Signs of Nuclear Spin Coupling in $^{11}\text{B}^{19}\text{F}_2$ ", J. Chem. Phys., <u>43</u> , 1841 (1965). (3) T. D. Coyle, J. J. Ritter, and T. C. Farrar, "Preparation and Properties of Difluoroborane", Proc. Chem. Soc., <u>25</u> , (1964). (4) T. D. Coyle, J. Cooper, and J. J. Ritter, "Preparation and Some Reactions of Difluoroborane", Inorg. Chem., <u>7</u> , 1014 (1968).

BF ₅ OSi	electric discharge rxn of BF ₃ in SiO ₂ reactor	infrared, mass spectrometry using full isotopic anal.	novel Si-O-B molecular structure suitable for bond angle and dissn. studies; unstable at r.t.	F. E. Brinckman and G. Gordon "Energetic Intermediates in Inorganic Synthesis: Reactions of Covalent Main Group Fluorides in Electric Discharges", Fourth Int. Symp. on Fluorine Chem., Estes Park, Colo., July 1967, Abstr. of Papers, p. 132.
BF ₂ OSiF ₃				
B ₂ F ₄	also studied as liquid	B ₂ Cl ₄ + SbF ₃ at -80°C	simplest polyboron fluoride; potential source of BF ₂ function in free radical reactions; B-B bond dissn. energy	(1) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Characterization of Transport Species in Electric Discharges", Proc. Int. Symp. on Decomposition of Organometallic Compounds to Refractory Ceramics, Metals, and Metal Alloys, Univ. Dayton Press (1967), p. 29.
diboron tetrafluoride				
tetrafluorodiborane-4		infrared, mass, NMR (11B, 19F) spectrometry		(2) F. E. Brinckman and G. Gordon, "Energetic Intermediates in Inorganic Synthesis: Characterization of Transport Species in Electric Discharges", Proc. Int. Symp. on Decomposition of Organometallic Compounds to Refractory Ceramics, Metals, and Metal Alloys, Univ. Dayton Press (1967), p. 29.
B ₂ F ₄ O	obsd. as parent-daughter ions	microwave discharge of BF ₃ in SiO ₂ reactor	novel B-O-B molecular structure suitable for bond angle and dissn. measurements; unstable even at low pressures	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. XII. Boron Trifluoride and Diboron Tetrafluoride", Inorg. Chem., <u>7</u> , 1742 (1968).
BF ₂ OBF ₂		direct inlet mass spectrometry, isotopic anal.		See refs. 1, 2 under BF ₅ OSi.

$n\text{B}_2\text{H}_6$	studied as liquid	$n\text{BF}_3 \cdot \text{O}(\text{C}_2\text{H}_5)_2 + \text{LiAlH}_4$ (n = 10 or 11)	infrared (1), NMR (^1H , ^{10}B , ^{11}B) spectrometry (2)	parent material for synthesis of selected isotope (^1H , ^{10}B , ^{11}B) boranes; de-tailed interpretation of NMR spectra	T. D. Coyle T. C. Farrar R. B. Johannesen	(1) W. J. Lafferty, A. G. Maki, and T. D. Coyle, "High Resolution Infrared Spectrum and Structure of Diborane", J. Mol. Spectroscopy, <u>33</u> , 345 (1970). (2) T. C. Farrar, R. B. Johannesen, and T. D. Coyle, "Magnetic Non-Equivalence in the High Resolution NMR Spectra of Diborane", J. Chem. Phys., <u>49</u> , 281 (1968).
diborane						See also $\text{C}_2\text{H}^{11}\text{BD}_6\text{O}_2$ in SECTION II.

CCl_3F	also studied as liquid, solid	commercial	infrared, NMR (^{19}F , ^{35}Cl , ^{37}Cl) spectrometry, volatility, m.p.	model compound for structure in liquid; anisotropic motions in liquids; angular momentum cross-sections	T. C. Farrar	
Freon-11						
CHN		$\text{NaCN} + \text{H}_2\text{SO}_4$	infrared and mass spectrometry	used for photoionization studies: parent compound for X-CN series	T. D. Coyle	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. IX. Hydrogen Cyanide and Acetonitrile", J. Chem. Phys., <u>48</u> , 4765 (1968).
HCN						
CH_3NO_2		$\text{NaNO}_2 + \text{CH}_3\text{OH} + \text{H}_2\text{SO}_4$	infrared, ultraviolet, mass spectrometry; vapor phase chromatography	prepd. for photoionization study	T. D. Coyle	
CH_3ONO						
methyl nitrite						

C_2HBCl_2		trans- $ClCH = CHBCl_2 + h\nu$	mass, infrared spectrometry; cleavage with propionic acid $\rightarrow C_2H_2 + HCl$	first ethynylboron chloride isolated; novel substituted acetylene; suitable for studies on coordination of saturation of B; air/moisture sensitive	J. J. Ritter, T. C. Coyle, and J. M. Bellama, "Synthesis of Ethynylboron Halides", Chem. Comm., 908 (1969).
ethynylidichloroborane					
C_2HBF_2	C_{2v} symmetry	$(CH_3)_3SnC \equiv CH + BF_3$ at $-80^\circ C$	mass, infrared, and microwave spectrometry, cleavage in glass with propionic acid $\rightarrow C_2H_2 + SiF_4$	first ethynylboron fluoride isolated; suitable for extensive structural anal.; air/moisture sensitive	See ref. under C_2HBCl_2 . W. J. Lafferty, J. J. Ritter, "Microwave Spectrum, Structure, and Dipole Moment of Ethynylidifluoroborane, $HC \equiv CBF_2$ ", Chem. Comm., 909 (1969).
ethynylidifluoroborane					
$ClFO_3$ $FClO_3$ perchloryl fluoride	also studied as liquid, solid	commercial	see CCl_3F	see CCl_3F	T. C. Farrar
$Cl_nF_{4-n}Si$ chlorofluorosilanes	$n = 1-3$ also studied as liquids	$SiCl_4 + SiF_4 + AlCl_3$ (cat.) at $150^\circ C$ (1) electric discharge in $SiCl_4 + SiF_4$ (2)	infrared, mass (full isotopic anal.), NMR [^{19}F , ($^{19}F\{^{29}Si\}$)] spectrometry; mol. volatility, phase chromatography	model compounds for structural, dipole moment ($n = 1, 3$), and magnetic resonance properties; synthetic intermediates	(1) R. B. Johannesssen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., 72, 660 (1968). (2) See refs. under BF_5OSi . (3) See ref. under F_4Si .

D	D(atoms)	microwave discharge in $D_2O(g)$	measurement of electron g-factor relative to g_J of H atoms	experimental	M. Linzer	M. Linzer, "Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deuterium and Atomic Nitrogen", Bull. Am. Phys. Soc., <u>12</u> , 507 (1967).
F_2HP		$PF_2I + HI$ (1)	mol. wt.; infrared NMR ($^1H, ^{19}F, ^{31}P$)(2) spectrometry	NMR relative sign measurements; reagent uses; air/moisture sensitive	T. D. Coyle	(1) R. W. Rudolph and R. W. Parry, "Fluorophosphine Ligands. I. The Preparation and Characterization of Difluorophosphine", Inorg. Chem., <u>4</u> , 1339 (1965). (2) R. B. Johannesen, "NMR Studies of Inorganic Fluorides. IV. Relative Signs of Coupling Constants in CH_3SiF_3 , $HSiF_3$, and HPF_2 ", J. Chem. Phys., <u>47</u> , 3088 (1967).
F_3OP	OPF_3	studied as liquids also	commercial; fractional distillation	related to study of electric discharge rxns in PF_3 (1); model compounds for study of structures and electron anisotropies (2)	F. E. Brinckman T. C. Farrar (2)	(1) F. E. Brinckman, and G. Gordon, "Formation of μ -Oxyfluorophosphines and Polyphosphines in Gaseous Discharge Reactions", 4th Middle Atlantic Regional Mtg of the Am. Chem. Soc., Wash. D. C., February 1969, Abstr. of Papers, p. 41.
F_3P	PF_3		infrared, mass NMR ($^{19}F, ^{31}P$)(2) spectrometry; volatility			
F_4Si	Also obsd. as liquid (1)	commercial; fractional distillation	infrared, mass, NMR (^{19}F) spectrometry; volatility, mol. wt., vapor phase chromatography	Si-F coupling constant; ^{29}Si NMR by double resonance	T. D. Coyle T. C. Farrar	T. D. Coyle, R. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "NMR Studies of Inorganic Fluorides. II. Solvent Effects on $J(^{29}Si-^{19}F)$ in SiF_4 ", J. Phys. Chem., <u>70</u> , 1682 (1966).
	SiF_4 tetrafluorosilane					

F_6Ge_2O (GeF_3) $_2O$	obsd as parent and daughter ions	microwave dis- charge rxns. of GeF_4 and $GeF_4 + SiF_4$ in SiO_2 reactors	direct inlet mass spectrometry, full isotopic anal.	candidates for molecular structure and bond dissn. energy measure- ments; yields very small	F. E. Brinckman	See ref. 2 under BF_5OSi .
F_6GeOSi GeF_3OSiF_3						
F_6Si_2	also studied as liquid	$Si_2OCl_6 +$ $SbF_3(1)$; electric discharge rxn of SiF_4 with $SiO_2(2)$	NMR [$^{19}F, (^{19}F\{^{29}Si\})$] (1); mass (2) spectrometry	suitable for determination of Si-O-Si bond angle; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) R. B. Johannesen, T. C. Farrar, F. E. Brinckman, and T. D. Coyle, "NMR Studies of Inorganic Fluorides. I. High-Resolution ^{19}F Spectra of Si_2F_6 and $(SiF_3)_2O$ ", J. Chem. Phys., 44, 962 (1966). (2) See ref. 2 under BF_5OSi .
Si_2OF_6 hexafluoro- disiloxane						
F_6Si_2	also studied as liquid	$Si_2Cl_6 +$ $SbF_3(1)$	NMR [$^{19}F, (^{19}F\{^{29}Si\})$] (1); spectrometry chemical reactivity (2)	parent com- pound for perfluoro- disilanyl chemistry; suit- able for detm. of Si-Si bond energy; purified by vapor phase chromatography; air/moisture sensitive	F. E. Brinckman	(1) See ref. 1 under F_6OSi_2 . (2) F. E. Brinckman, T. D. Coyle, and L. Fishman, "Forma- tion of a Perfluoro- disilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Abstr. of Papers, p. D14.
Si_2F_6 hexafluorodisilane						
Ga_2O		$Ga_2O_3(s)$ heated to 1200-1500°C	mass spectrometry		R. C. Paule	
Ga_2O_4W Ga_2WO_4		$Ga_2O_3(s) +$ W heated to 1200°C	mass spectrometry		R. C. Paule	
N N(atoms)		microwave discharge in $N_2(g)$	see D	see D	M. Linzer	See ref. under D.

II. LIQUIDS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION		REFERENCES (Author, Title, Journal)
BD _{3-n} F ₃ H ₃ P F ₃ P·BH _n D _{3-n}	n = 1-3 (2)	PF ₃ +B ₂ H ₆ (deuterated)	synthesis, microwave spectrometry	rotational spectrum, barrier to internal rotation	T. D. Coyle	R. Kuczkowski and D. Lide, "Microwave Spectrum, Structure, Dipole Moment, and Barrier to Internal Rotation of Phosphorus Trifluoride-Borane", J. Chem. Phys., <u>46</u> , 357 (1967).	
BF ₇ Si ₂ SiF ₃ SiF ₂ BF ₂		prepd. at Rice Univ. (a) SiF ₂ (g) + BF ₃ (g) (b) co- condensed at -196°C	infrared, NMR (11B, 19F) spectro- metry	series of novel Si-B compounds; qualitative detm. of structure; air/moisture sensitive	F. E. Brinckman T. D. Coyle T. C. Farrar	P. L. Timms, T. C. Ehlert, J. A. Margrave, F. E. Brinckman, T. C. Farrar, and T. D. Coyle, "Silicon- Fluorine Chemistry. II. Silicon-Borane Fluorides, J. Am. Chem. Soc., <u>87</u> , 3819 (1965).	
BF ₉ Si ₃ n-Si ₃ F ₇ BF ₂							

B_2Cl_4	unstable liquid	electric discharge reduction of BCl_3 (1)	infrared, mass (2) spectrometry; chemical reactivity (3)	parent material for chemistry of diboron compounds; detm. of B-B bond energy	J. J. Ritter	(1) T. Wartik, et al., "Diboron Tetrachloride", Inorg. Syn. <u>10</u> , 118 (1967); (2) V. H. Dibeler and J. A. Walker, "Mass Spectrometric Study of Photolonization. XIII. Boron Trichloride and Diboron Tetrachloride", Inorg. Chem., <u>8</u> , 50 (1969); (3) T. D. Coyle and J. J. Ritter, "Reactions of Diboron Tetrahalides with Some Unsaturated Organometallic Compounds", J. Organomet. Chem., <u>12</u> , 269 (1968).
BrF_3 -n bromofluoro-silanes	n = 1-3 studied as liquids and gases	SiF_4 + $SiBr_4$ + $AlBr_3$ catalyst at 150°C	infrared, mass spectrometry; NMR [^{19}F , (^{29}Si)] (1,2) spectrometry; volatility	dipole moments detmd. for SiF_3Br and $SiBr_3F$ (with A. Marryot, NBS)	F. E. Brinckman T. D. Coyle	(1) T. D. Coyle, R. B. Johannesen, F. E. Brinckman, and T. C. Farrar, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. II. Solvent Effects on J(^{29}Si - ^{19}F) in Silicon Tetrafluoride", J. Phys. Chem., <u>70</u> , 1682 (1966). (2) R. B. Johannesen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., <u>72</u> , 660 (1968).

$\text{CH}_3\text{P}_2\text{Cl}_3$ methyltrichloro- diborane-4	unstable	$\text{B}_2\text{Cl}_4 + (\text{CH}_3)_4\text{M}$ (M = Ge, Sn, or Pb)	infrared, mass spectrometry; cleavage rxns. with $\text{Ag}_2\text{O} + \text{NH}_3(\text{aq}) \rightarrow \text{CH}_4 + \text{H}_2$	unsymmetrical diboron halide; useful for studies of additions to olefinic and acetylenic systems; air/moisture sensitive	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Recent Studies in Organoboron Chemistry. Some Chemical Consequences of the Boron Subhalides", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. R1.
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$^{13}\text{CH}_3\text{I}$ $^{13}\text{CH}_4\text{O}$ $^{13}\text{CH}_3\text{OH}$ [^{13}C]-methanol	also studied as gas	commercial, high-vacuum degassed	infrared, NMR (^1H , ^{13}C) spectrometry; m. p.	model compounds for study of ^{13}C T_1 and T_2 values	T. C. Farrar	See also $\text{C}_2\text{H}_4\text{O}_2$.
$\text{C}_2\text{H}^n\text{BD}_6\text{O}_2$ (CD_3O) $_2$ ^nBH	n = 10, 11; also studied as solid	$\text{CD}_3\text{OH} + \text{nB}_2\text{H}_6$	infrared, NMR (^1H , ^{11}B) spectrometry; mol. wt.; m. p.	isotopic composition provides selected nuclear spin properties only example of B-H bond distance measurement in 3-coordinate boron compounds; motional studies; precursors of HBF_2	T. D. Coyle T. C. Farrar	(1) T. C. Farrar, J. Cooper, and T. D. Coyle, "Proton Broad-Line NMR Study of [$^{12}\text{H}_6$]dimethoxy-[^{11}B]borane", Chem. Comm., 610 (1966). (2) N. Boden, H. S. Gutowsky, J. R. Hansen, and T. C. Farrar, "Nuclear Magnetic Relaxation Studies of (CD_3O) $_2\text{BH}$ ", J. Chem. Phys., 46, 2849 (1967). (3) T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, 73A, 195 (1969).

$C_2H_2B_2Cl_4$	trans-(BCl_2)CH=CH(BCl_2)	cis-(BCl_2) $_2C_2H_2$ + $h\nu$	infrared, mass, NMR spectrometry; cleavage rxn of deuterio-compound with $Ag_2O + NH_3(aq)$ → trans-dideuteroethylene	established the mode of addition of B_2Cl_4 to C_2H_2 ; pointed up the utility of 1H NMR while irradiating at the boron frequency in distinguishing between 1,1 and 1,2 isomers while demonstrating the dangers of assigning structures on the basis of J_{H-H} ; demonstrated utility of $Ag_2O/NH_3(aq)$ as B-C cleaving agent with retention of configuration; air/moisture sensitive	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Structure, Isomerization, and Cleavage of 1,2-Bis-(dichloroboryl)-ethylene", J. Am. Chem. Soc., <u>89</u> , 5739 (1967).
$C_2H_2B_4Cl_4$ 1,1,2,2-tetrakis(dichloroboryl)ethane	white solid, melts at 30°C	$ClCH=CHBCl_2 + 2B_2Cl_4$ or $HC\equiv CH + 2B_2Cl_4$	elemental anal. mol. wt., rxn. ratios	further confirmation of the reaction between B_2Cl_4 and halo-olefins to produce a known compound; air/moisture sensitive	J. J. Ritter T. D. Coyle	(1) J. J. Ritter, T. D. Coyle, J. M. Bellama, "The Inter-action of B_2Cl_4 with Halo-olefins", Abstracts, 156th National Meeting of Amer. Chem. Soc., Atlantic City, N. J., (1968) p. INOR 166; (2) C. Chambers, A. K. Holliday, and S. M. Walker, "1,2-Tetrakis(dichloroboryl)-ethane", Proc. Chem. Soc., 286 (1964).

$C_2H_3B_2Cl_2MX_3$ ($M=C, Si, Ge, Sn$; $X=Cl, CH_3$)	involatile liq. or oils	$B_2Cl_4 + CH_2 =$ $CHMX_3$	elemental anal.; mol. wt., rxn. ratios	provides series of model com- pounds for structure- reactivity relationships in vicinal- and geminal- substituted organo- metal systems	J. J. Ritter T. D. Coyle	See ref. (3) under B_2Cl_4 .
$C_2H_3B_3Cl_6$ 1,1,2-tris(dichloro- boryl)-ethane		$CH_2 = CHCl +$ $2B_2Cl_4$ or $CH_2 = CHBCl_2$ $+ B_2Cl_4$	complete elemental anal.; mol. wt.; infrared, NMR spectrometry, rxn. ratio	demonstrated a reaction pathway be- tween B_2Cl_4 and halo olefins contrary to earlier reports of no reaction; suggested "BCl" as a possible "reactive inter- mediate" respon- sible for the observed product; air/moisture sensitive	J. J. Ritter T. D. Coyle	(1) See ref. under $C_2H_3B_2Cl_2MX_3$. (2) See ref. (1) under $C_2H_2B_4Cl_4$.
$C_2H_4O_2$ $CH_3^{13}COOH$ $\alpha[^{13}C]$ -acetic acid		commercial; high-vacuum degassed	infrared, NMR ($^1H, ^{13}C$) spectro- metry; m.p.	model com- pound for study of ^{13}C T_1 and T_2 values	T. C. Farrar	See also $^{13}CH_3I$ and $^{13}CH_4O$.

$C_3H_{10}F_3NSi_2$ $(CH_3)_3SiNHSiF_3$	$[(CH_3)_3Si]_2NH + SiF_4$	infrared, mass, NMR (1H , ^{19}F) spectrometry, elemental anal., subsequent rxns.	F. E. Brinckman	model compound for preparative route to perfluorosilazanes	See ref. 2 under BrF_3-nSi .
$C_3H_{10}F_5NSi_3$ $(CH_3)_3SiNHSi_2F_5$	$[(CH_3)_3Si]_2NH + Si_2F_6$			model compound for derivative chemistry of perfluoropolysilanes based cleavage of Si-N	F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluoro-disilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. D14.
C_4HCoO_4 $HCo(CO)_4$	studied as solid; liquid unstable at $> -45^\circ C$ see ref. (1)	NMR (1H) spectrometry (1,2)	F. E. Brinckman T. D. Coyle T. C. Farrar	model compound for application of NMR in detm. metal-H bond distances; basis for reexamination of basic theory; see C_5HMnO_5	(1) T. C. Farrar, F. E. Brinckman, T. D. Coyle, A. Davidson and J. W. Faller, "A Broad-Line Proton Magnetic Resonance Study of Cobalt Tetra-carbonyl Hydride", Inorg. Chem., <u>6</u> , 161 (1967). (2) D. L. VanderHart, H. S. Gutowsky, and T. C. Farrar, "Dipole-Dipole Interactions of a Spin 1/2 Nucleus with a Quadrupole-Coupled Nucleus", J. Am. Chem. Soc., <u>89</u> , 5056 (1967).
$C_4H_2FeO_4$ $H_2Fe(CO)_4$	studied as liquid	infrared, NMR (1H) spectrometry	F. E. Brinckman T. C. Farrar	candidate compound for Fe-H distance detm., J_{Fe-H}	See C_5HMnO_5 .

$C_4H_9B_3$ C, 3-dimethyl-1,2-dicarbaclovo-pentaborane(5)	also studied as solid	prepared at Mass. Inst. Tech.; and at NBS by F. E. Brinckman	infrared, NMR (1H) spectro-metry	NMR (1H , ^{11}B) spectrometry	clarification of molecular structure	T. C. Farrar	R. N. Grimes, "A New Isomer of $C_2B_3H_5$, C, 3-Dimethyl-1,2-dicarbaclovo-pentaborane(5)", J. Am. Chem. Soc., <u>88</u> , 1070 (1966).
C_5HMnO_5 $HMn(CO)_5$	also studied as solid				model compound for application of NMR in detm. metal-H bond distance; see also C_4HCoO_4	T. C. Farrar	T. C. Farrar, Sister W. Ryan, A. Davidson, and J. W. Faller, "Manganese-Hydrogen Bond Distance in $HMn(CO)_4$ ", J. Am. Chem. Soc., <u>88</u> , 184 (1966).
C_5HReO_5 $HRe(CO)_5$							
$C_5H_{15}N_3Si$	n = 3	(a) $CH_3N_3 + CH_3MgCl$ (b) a + $(CH_3)_4SiCl_4$ -n at -40 to -80°C; see ref. (1)	infrared, NMR (1H) spectro-metry (1,2), elemental anal., mol. wt.		parent compounds for synthesis of 1,3-dimethyl-triazeno-metal(lolid) deriv.; novel bidentate ligand for coordination studies; air/moisture sensitive	F. E. Brinckman	(1) F. E. Brinckman, H. S. Haiss, and R. A. Robb, "Metal-Nitrogen Bonding, Covalent Complexes of 1,3-Dimethyltriazene with Elements of Groups I, II, III, IV, and V", Inorg. Chem., <u>4</u> , 936 (1965). (2) F. E. Brinckman, K. Nelson, R. Barefoot, and H. Haiss, "Silicon-Nitrogen Bonding. Penta- and Hexa-coordinated Inter-mediates in Methyl-silazanes", in preparation.
$C_6H_{18}N_6Si$ $(CH_3)_nSi(CH_3N=NCH_3)_{4-n}$	n = 2						

C ₆ H ₆	studied also as solid	commercial	NMR (¹ H, ¹³ C) spectrometry	application of Fast- Fourier trans- form NMR spectrometry to natural abundance ¹³ C	T. C. Farrar	See also C ₆ H ₆ I under SECTION IV.
benzene						
C ₆ H ₁₂						
cyclohexane		commercial, spectro- grade	NMR (¹ H) spectrometry	measurement of ¹ H g- factor rela- tive to g _J of H atoms; secondary standard for g-factor detms; check on cal- culation of shielding para- meter for H ₂ molecule	M. Linzer	M. Linzer, "Measure- ment of the Proton g-factor in Cyclo- hexane and the Elec- tron g-factors in Atomic Deuterium and Atomic Nitrogen", Bull. Am. Phys. Soc., 12, 507 (1967).
Cl ₄ V	obsd. as neat liquid and in TiCl ₄ soln.; also obsd. as poly- crystalline solid	commercial, fractionally distilled	EPR spectrometry (as a function of frequency, temp.); magnetic suscepti- bility between 78-307°K; optical spectrophotometry of VCl ₄ in TiCl ₄	detm. magnetic parameters, Jahn-Teller distortion; paramagnetic relaxation time, T ₁ ; air/moisture sensitive	R. B. Johannesen	R. B. Johannesen, G. A. Candela, and T. Tsang, "Jahn- Teller Distortion: Magnetic Studies of Vanadium Tetra- chloride", J. Chem. Phys., 48, 5544 (1968).
Cl ₅ FSi ₂		Si ₂ Cl ₆ + SiF ₄ + AlCl ₃ (cat.) at 150°C	infrared, mass, NMR [¹⁹ F, (¹⁹ F{ ²⁹ Si})] spectrometry	first ex- ample of mixed per- halogenated disilane; ¹⁹ F "tag" permits double- resonance NMR of Si nuclei	F. E. Brinckman T. D. Coyle	See ref. 2 under Br _n F _{3-n} Si.
Si ₂ Cl ₅ F pentachlorofluoro- disilane						R. B. Johannesen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Some Fluorinated Mono- and Polysilanes", 153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers, p. 1113.

Cl ₆ Si ₂	commercial, fractional distillation; electric discharge in SiCl ₄	infrared, mass (full isotopic anal.), spectro-metry; vapor phase chromatography; volatility, cleavage rxn with H ₂ O → H ₂ +Cl-	parent material for synthesis of compounds bearing Si-Si bond; suitable for Si-Si bond energy detm. air/moisture sensitive	F. E. Brinckman	F. E. Brinckman, J. Cooper, and T. D. Coyle, "Interactions of Some Halosilanes with Nitrogen Bases", 153rd Natl. Mtg. Am. Chem. Soc., Miami Beach, Fla., April 1967, Abstr. of Papers, p. L112.
					See SECTION I under F ₆ Si ₂ .

H ₂ O	polymeric	not available	infrared, NMR (¹ H) spectrometry	attempt to establish nature of protons in sample (work with M. Malmberg, NBS)	T. C. Farrar E. D. Lippincott
"polywater"					

III. GLASSES

NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION							REFERENCES (Author, Title, Journal)
COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS			
barium aluminoboro- fluorosilicate glasses; example: Al ₂ O ₃ 12 mol% B ₂ O ₃ 16 BaF ₂ 20 BaO 8 SiO ₂ 44	microspheres, 5-50 μ dia.	melting, chill- ing, size reduction, flame spheriz- ing	refractive index, thermal expansion, sphere size	filler mat- erial for dental composites	G. W. Cleek	R. L. Bowen and G. W. Cleek, "X-Ray Opaque Reinforcing Fillers for Composite Mat-erials", J. Dental Res., 48, 79 (1969).	
glasses from Al ₂ O ₃ -BaO-SiO ₂ system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek		
aluminosilicate glass, alkali- free	NBS Standard Reference Material # 715	melting, chilling	Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	ASTM draft in preparation.	
glasses from B ₂ O ₃ -BaO-SiO ₂ system	glass blocks	melting, annealing	chemical analysis; refractive index; density; liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	E. H. Hamilton, G. W. Cleek, and O. H. Grauer, "Some Properties of Glasses in the System Barium Oxide-Boric Oxide- Silica", Amer. Ceram. Soc., 41, 209 (1958).	
glasses from B ₂ O ₃ -Ln ₂ O ₃ system	small fragments	gravity separated from two immiscible liquids	polarizing microscopy	high-index solid state	E. M. Levin	E. M. Levin, "Liquid Immiscibility in the Rare Earth Oxide- Boric Oxide Systems", Phys.& Chem. Glasses, 7, 90 (1966).	

specimens used for theoretical studies of immiscibility

W. Haller

W. Haller, D. H. Blackburn, F. E. Wagstaff, and R. J. Charles, "The Metastable Immiscibility Surface in the System $\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{SiO}_2$ ", J. Am. Ceram. Soc. 53, 34 (1970).

domain development kinetics; critical immiscibility temp.

melting, chilled

glass blocks

glasses from $\text{BaO}_3-\text{Na}_2\text{O}-\text{SiO}_2$ system

tested as possible method to detm. neutron flux

D. H. Blackburn

"Glass Beads for Neutron Flux Measurements", NBS Technical Highlights, 1965, p. 86

neutron activation anal.

melting, chilling, grinding

glass prisms

^{10}B -enriched borate glasses doped with Co, Dy, or In

specimens may be used for detm. of other properties

G. W. Cleek

refractive index, transmittance, liquidus temp.

melting, annealing

glass blocks

glasses from $\text{BaO}-\text{La}_2\text{O}_3-\text{SiO}_2$ system

specimens are being evaluated for fluorescence lifetime

G. W. Cleek

E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Transfer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", J. Appl. Phys. 41, 364 (1970).

evaluation for laser use

melting, chilling, annealing

blocks 3 x 3 x 3 cm.

a base glass: BaO 5 mol% Na_2O 15 SiO_2 75 ZnO 5 doped with various rare earths: Eu, Er, Nd

specimens may be used for detm. of other properties

G. W. Cleek

refractive index, transmittance, liquidus temp.

melting, annealing

glass blocks

glasses from $\text{BaO}-\text{Nb}_2\text{O}_5-\text{SiO}_2$ system

specimens may be used for detm. of other properties

G. W. Cleek

refractive index, transmittance, liquidus temp.

melting, annealing

glass blocks

glasses from $\text{BaO}-\text{SiO}_2-\text{TiO}_2$ system

glasses from BaO-SiO ₂ -ZnO system	glass blocks	melting, annealing	refractive index, transmittance, liquidus, temp.	specimens may be used for detm. of other properties	G. W. Cleek
alkali-lime- silica glass: CaO 11.6 mol% K ₂ O 7.7 Na ₂ O 8.7 SiO ₂ 70.5	NBS Standard Reference Material # 710	melting, chilling	viscosity by beam bending	not NBS certified for this method but suitable as standard	A. Napolitano
			surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano
			viscosity by penetrometer	not NBS certified for this method but suitable as standard for ASTM flow point detm.	(4) J. E. Kelley, T. D. Robert, and H. M. Hanes, "A Penetro- meter for Measuring the Absolute Viscosity of Glass", U. S. Bureau of Mines RI 6358 (1964).
Fe-containing silicate glasses	30 x 30 x 2 mm	melting, chilling	Mossbauer spectrometry	specimens covering a wide range of Fe content may be used for characterization by Mossbauer spectroscopy	G. W. Cleek

alkali-lead-silica glass: K ₂ O 5.6 mol% Na ₂ O 2.5 PbO 45.3 SiO ₂ 46.0	NBS Standard Reference Material # 711	melting, chilling	surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano	See ref. 2 under alkali-lime-silica glass.
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	ASTM draft in preparation.
glasses from Na ₂ O-SiO ₂ -TiO ₂ system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other glass properties	G. W. Cleek	E. H. Hamilton and G. W. Cleek, "Properties of Sodium Titanate Silicate Glasses", J. Res. NBS, <u>61</u> , 89 (1958).
porous silica glass SiO ₂	powders, 50-200 mesh particles	melting, chilling, thermal domain development, differential dissolution	mercury intrusion porosimetry, nitrogen absorption	possible surface area and porosity standards	W. Haller	(1) W. Haller, "Rearrangement Kinetics of the Liquid-Liquid Immiscible Microphases in Alkali-Boro-silicate Melts", J. Chem. Phys., <u>42</u> , 686, (1965). (2) N. M. Winslow and J. J. Shapiro, "An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration", ASTM Bull, <u>39</u> (Feb. 1959).
various	glass prisms	melting, chilling, annealing, polishing	refractive index	prisms covering range of refractive indices 1.5 to 1.9 measured by minimum deviation method for NaCl, Na ₂ CO ₃ , and NaF	G. W. Cleek	

IV. SINGLE CRYSTALS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Al ₂ O ₃ alumina	also studied as powder, see SECTION V	(1) commercial powder and crystals (2) purified powder by solar furnace	melting point	melting point detm. for SRM	S. J. Schneider	S. J. Schneider and C. L. McDaniel, "Effect of Environment upon the Melting Point of Al ₂ O ₃ ", J. Res. NBS, <u>71A</u> , 317 (1967).
	single crystal bar	commercial	compressibility to 10Kb	see also under BH ₃ O ₃ , SECTION V	C. E. Weir*	S. J. Schneider, "Cooperative Determination of the Melting Point of Al ₂ O ₃ ", Pure and Applied Chemistry, to be published.
	single crystals and bicrystals, 3 x 8 x 20 mm	chemical vapor deposition, AlCl ₃ + [O] → Al ₂ O ₃ at 1700-1750°C	spectrochemical and activation anal., optical microscopy, x-ray diffractometry	material of high physical perfection and chemical purity; suitable for diffusion studies, physical properties	H. S. Parker	C. E. Weir, "Compressibility of Eleven Inorganic Materials", J. Res. NBS, <u>69A</u> , 29 (1965).
pure and with following dopants: Cr, Mg, Mg+Cr, Ti+Cr, Ti, V, Mn, Mn+Cr, Zn +Cr, Fe+Cr	~ 1cm	flame fusion, prepd. by D. E. Roberts	optical, EPR spectrometry	laser properties (optical and x-ray damage)	R. F. Blunt T. Chang	H. S. Parker and C. A. Harding, "Vapor Growth of Aluminum Oxide Bicrystals", Abstracts, ACCG-NBS Conference on Crystal Growth, Aug. 11-13, 1969, Washington, D. C., p. 42.
						C. M. Stickley, et al, "Color Centers and Ruby-Laser Output-Energy Degradation", J. Appl. Phys., <u>40</u> , 1792 (1969).

$\text{Al}_{1-n}\text{Cr}_n\text{K}_2\text{O}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$ $n = 1$ to 0.02 $\text{KAl}_{1-n}\text{Cr}_n(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	grown from aq. soln.	optical, magnetic	ferro- electric, see also $\text{CrH}_4\text{NO}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$	G. A. Candela S. E. Stokowski*
AsGa GaAs gallium arsenide	highly doped and compensated	vapor phase epitaxy on GaAs substrate, GaCl_3 + AsCl_3 →; prepared by Monsanto Co.	spectral examination of temp. dependence of radiative recombination induced by electrical injection across p-n junction or by optical excitation	understanding A. H. Neill* of band N. Winogradoff* structure of GaAs responsible for unusual absorption- edge exhibited; GaAs semi- conductor injection lasers, im- provement of room temp. performance; present efficient use of GaAs as a laser necessitates cryogenic condi- tions, cause of large decrease in efficiency at room temp. not fully understood, such studies should lead to specific material useful for improved laser performance
				(1) N. N. Winogradoff and H. Kessler, "White Emission and Electrical Characteristics of Epitaxial GaAs Lasers and Tunnel Diodes", Solid State Comm., 2, 119 (1964). (2) N. N. Winogradoff, K. Owen and R. Cutnutt "The Radiative Band Pinch Effect and Temperature Dependence of Radiative Recom- bination in GaAs", Int. J. Electronics, 22, 229 (1967). (3) N. Winogradoff, "Field Control of the Quantum Efficiency of Radiative Recombi- nation in Semicon- ductors", Phys. Rev., 138A, 1562 (1968). (4) N. Winogradoff and A. H. Neill, "Band Tailing Effects and the Temperature De- pendence of Radiation Recombination in Compensated Epitaxial GaAs Laser Junctions", IEEE J. Quant. Electronics, 4, 111 (1968).

B ₂ BaO ₄ BaO·B ₂ O ₃	small, tenths of mm	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high-temp form, meta- stable at room temp.	A. Mighell A. Perloff	A. D. Mighell, A. Perloff and S. Block, "The Crystal Structure of the High Temper- ature Form of Barium Borate, BaO·B ₂ O ₃ ", Acta Cryst., <u>20</u> , 819 (1966).
B ₂ BiO ₄ 2Bi ₂ O ₃ ·B ₂ O ₃	~ 0.1 mm range	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.		A. Perloff	A. Perloff, paper in prepn. for submission to Acta Cryst.
B ₄ O ₇ Pb PbO·2B ₂ O ₃	~ 0.1 mm range	crystallized from melt of stoichiometric composition	unit cell and diffraction symbol from single crystal data	structure by analogy to isomorphous SrO·2B ₂ O ₃	A. Perloff	A. Perloff and S. Block, "The Crystal Structure of the Strontium and Lead Tetraborates, SrO· 2B ₂ O ₃ and PbO· 2B ₂ O ₃ ", Acta Cryst., <u>20</u> , 274 (1966).
B ₄ O ₇ Sr SrO·2B ₂ O ₃	needles; length ~ 0.1 mm, cross- section ~ .01 mm	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.	see B ₄ O ₇ Pb	A. Perloff	See ref. under B ₄ O ₇ Pb.
4B ₂ O ₃ ·BaO BaB ₈ O ₁₃	twinned crystal, 1 x 1 x 2 mm	melt solidi- fication, required seeding for crystallization	spectrochemical anal.; optical, x-ray powder diffractometry (from ambient to 850°C); differ- ential thermal anal.	piezo- electric at ambient temp. (ortho- rhombic form); transforms reversibly to tetragonal form at 700°C	C. R. Robbins E. M. Levin	C. R. Robbins and E. M. Levin, "Phase Transformation in Barium Tetraborate", J. Res. NBS, <u>73A</u> , 615 (1969).
4B ₂ O ₃ ·Na ₂ O Na ₂ O·4B ₂ O ₃	small (~ 0.1 mm range)	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high temp. form, meta- stable at room temp.	A. Perloff	A. Hyman, A. Perloff, F. Mauer, and S. Block, "The Crystal Structure of Sodium Tetraborate", Acta Cryst., <u>22</u> , 815 (1967).

BaGe ₄ O ₉	1 x 1 x 3 mm	melt solidi- fication of stoichiometric composition	optical and x-ray diffractometry (1), including single crystal structure detm. (2)	type structure for AB ₄ O ₉ ger- manates, see also Ge ₄ O ₉ Pb and Ge ₄ O ₉ Sr in SECTION V	C. R. Robbins	(1) C. R. Robbins and E. M. Levin, "Tetra- germanates of Strontium, Lead and Barium of Formula Type AB ₄ O ₉ ", J. Res. NBS, <u>65A</u> , 127 (1961). (2) C. Robbins, A. Perloff, and S. Block, "Crystal Structure of BaGe [Ge ₃ O ₉] and its Relation to Benitoite", J. Res. NBS, <u>70A</u> , 385 (1966).
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Ba ₂ Ge ₂ O ₈ Ti Ba ₂ TiGe ₂ O ₈	1 x 1 x 3 mm	melt solidi- fication and melt solidi- fication using TiO ₂ as flux (1)	spectrochemical; optical and x-ray powder diffracto- metry; differential thermal anal.	crystals exhibit pronounced domain struc- ture, show very strong piezoelectric response (1); phosphor (2)	C. R. Robbins	(1) Work in progress with particular in- terest in the crystal chemistry, domain structure and piezo- electric property of the compound. (C. R. Robbins, 1969). (2) G. Blasse, "Fluo- rescence of Compounds with Fresnoite (Ba ₂ TiSi ₂ O ₈) Structure", J. Inorg. Nucl. Chem., <u>30</u> , 2283 (1968).
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Ba ₂ O ₈ Si ₂ Ti Ba ₂ TiSi ₂ O ₈	1 x 1 x 5 mm	melt solidi- fication using TiO ₂ as flux (1)	spectrochemical; optical, x-ray powder diffracto- metry (powder and single crystal), differential thermal anal. (1)	piezoelectric (1); phosphor (2); type structure	C. R. Robbins	(1) C. R. Robbins, "Growth of Fresnoite (Ba ₂ TiSi ₂ O ₈) From a TiO ₂ Flux and Relation to the System BaTiO ₃ - SiO ₂ ", J. Res. NBS, <u>74A</u> , 229 (1970). (2) See ref. under Ba ₂ Ge ₂ O ₈ Ti.
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$\text{Be}_2\text{CuO}_{10}\text{Si}_2\text{Y}_2$ $\text{CuY}_2\text{Be}_2\text{Si}_2\text{O}_{10}$ copper yttrium gadolinite	light blue, 1 mm; also studied as poly- crystalline material	slow cooling from Na_2WO_4 or $\text{Na}_2\text{W}_2\text{O}_7$ flux	precession and x-ray powder diffraction anal.	see under $\text{Be}_2\text{Ln}_2\text{MO}_{10}\text{Si}_2$ (M = Mg, Ni, Zn, Co, Cu, Fe, Mn, Cd), SECTION V	H. S. Peiser	J. Ito, "The Synthesis of Gado- linite", Proc. Jap. Acad., <u>41</u> , 404 (1965). J. Ito, "A Note on the Gadolinite Syn- thesis", Proc. Jap. Acad., <u>42</u> , 634 (1966). J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., <u>52</u> , 1523 (1967).
$\text{Be}_2\text{O}_4\text{Si}(\text{V}^{+4})$ Be_2SiO_4 doped with V^{+4}	bright blue, 2 mm	slow cooling from V_2O_5 or $\text{Ba}_3(\text{VO}_4)_2$ flux	precession and x-ray powder diffraction anal.		H. S. Peiser	C. Frondel and J. Ito, "Synthesis of the Scandium Ana- logues of Beryl", Amer. Mineral., <u>53</u> , 943 (1968).
$\text{Bi}_{12}\text{GeO}_{20}$ $\text{GeBi}_{12}\text{O}_{20}$		see $\text{Bi}_{12}\text{MO}_{20}$	optical	see $\text{Bi}_{32}\text{Ga}_2\text{O}_{51}$	A. Feldman D. Horowitz	
$\text{Bi}_{12}\text{MO}_{20}$ $6\text{Bi}_2\text{O}_3 \cdot \text{MO}_2$	M = Ge, Si, Ti; 0.5 x 1 cm	melt grown	see also $\text{Bi}_{12}\text{GeO}_{20}$, $\text{Bi}_{32}\text{Ga}_2\text{O}_{51}$	Faraday notation; piezoelectric electro-optic	W. S. Brower	E. L. Venturini, E. G. Spencer and A. A. Ballman, "Plasto- Optic Properties of $\text{Bi}_{12}\text{GeO}_{20}$, $\text{Bi}_{12}\text{SiO}_{20}$, $\text{Sr}^x\text{Ba}_{1-x}\text{Nb}_2\text{O}_6$ ", J. Appl. Phys., <u>40</u> , 1622 (1969).
$7\text{Bi}_2\text{O}_3 \cdot \text{ZnO}$	0.5 x 1 cm	melt grown			W. S. Brower	
$17\text{Bi}_2\text{O}_3 \cdot \text{Ga}_2\text{O}_3$	0.5 x 1 cm	melt grown			W. S. Brower	
$\text{Bi}_{32}\text{Ga}_2\text{O}_{51}$ $16\text{Bi}_2\text{O}_3 \cdot \text{Ga}_2\text{O}_3$		pulled from melt, prepd. by W. S. Brower	optical	optically active, high Faraday ro- tation; see also $\text{Bi}_{12}\text{GeO}_{20}$	A. Feldman D. Horowitz	

Br ₂ bromine	from liquid at 20°C, 5Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	C. E. Weir, G. J. Piermarini, and S. Block, "Crystallography of Some High Pressure Forms of C ₆ H ₆ , CS ₂ , Br ₂ , CCl ₄ , and KNO ₃ ", J. Chem. Phys., 50, 2089 (1969)
CCl ₄	I structure from liquid at 20°C, 1Kb II structure from CCl ₄ -I at 80°C, 8Kb III structure from CCl ₄ -I at 120°C, 10Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .
CS ₂	frozen from liquid at 20°C, 12Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .
C ₅ FeN ₅ Na ₂ O·2H ₂ O Na ₂ Fe(CN) ₅ NO·2H ₂ O sodium nitroprusside	1 x 1 x 3 in. controlled growth from aq. soln.	Mossbauer spectroscopy	crystal structure, Standard Reference Material #725; Mossbauer spectroscopy	A. T. Horton	J. J. Spijkerman, D. K. Snediker, F. M. Ruegg, and J. R. DeVoe, "Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds", NBS Misc. Publ. 260-13 (1967). "Standard Reference Material for Mossbauer Spectroscopy", NBS Tech. News Bull., 50, 73 (May 1966).
C ₆ H ₆	I structure, see also SECTION II frozen from liquid at 20°C, 0.6 Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br ₂ .

$C_{10}H_{10}Cl_2CuN_2O_2$ Cu(C ₅ H ₅ NO) ₂ Cl ₂ with the following dopants: Ba, Cd, Ni, 61Ni (0.1-2%), Pb, and Zn bis(pyridine-N-oxide)copper (II) chloride	1 x 1 x 1 to 5 x 5 x 5 mm	grown from methanol soln.	optical and EPR spectrometry; chemical anal.	metal-metal interaction study	G. F. Kokoszka R. W. Duerst and G. F. Kokoszka, "Hyperfine Fields in Dimeric Coordination Complexes", J. Chem. Phys., <u>51</u> , 1673 (1969).
$C_{10}H_{10}Fe$ (C ₅ H ₅) ₂ Fe ferrocene	1 x 4 in	melt solidification	infrared and Raman spectrophotometry	infrared and Raman spectra vibrational transitions	A. T. Horton
$C_{12}H_{12}N_{10}NiO_6 \cdot 2H_2O$ Ni(C ₆ H ₆ N ₄) ₂ (H ₂ O) ₂ (NO ₃) ₂		C ₆ H ₆ N ₄ (2,2'-biimidazole) + Ni(NO ₃) ₂ + H ₂ O	single crystal x-ray anal.	study of hydrogen bonding between coordinated water and biimidazole molecules with nitrate groups	A. D. Mighell, C. W. Reimann, and F. A. Mauer, "The Crystal and Molecular Structure of Diaquobis(2,2'-biimidazole) Nickel (II) Dinirate, Ni(C ₆ H ₆ N ₄) ₂ (H ₂ O) ₂ (NO ₃) ₂ ", Acta Cryst., <u>B25</u> , 60 (1969).
$C_{12}H_{16}Br_2N_8Ni$ (C ₃ H ₄ N ₂) ₄ NiBr ₂ tetrakis (pyrazolo)-nickel (II) bromide		C ₃ H ₄ N ₂ -(pyrazole) + NiBr ₂ + H ₂ O	x-ray anal., single crystal structure detm.; optical spectrophotometry	role of internal hydrogen bond in detm. electronic energy levels of Ni ⁴⁺	A. D. Mighell, C. W. Reimann, and A. Santoro, "The Crystal and Molecular Structure of Di-bromotetrapyrazole Nickel (II)", Acta Cryst., <u>B25</u> , 595 (1969).
$C_{12}H_{16}Cl_2N_8Ni$ (C ₃ H ₄ N ₂) ₄ NiCl ₂ tetrakis(pyrazolo)-nickel (II) chloride		C ₃ H ₄ N ₂ (pyrazole) + NiCl ₂ + H ₂ O			C. W. Reimann, A. D. Mighell, and F. Mauer, "The Crystal and Molecular Structure of Tetrakispyrazole-nickel Chloride, Ni(C ₃ H ₄ N ₂) ₄ Cl ₂ ", Acta Cryst., <u>23</u> , 135 (1967).

$C_{12}H_{18}$ $C_6(CH_3)_6$ hexamethyl- benzene	1" dia. x 3"	melt solidi- fication	x-ray, neutron diffractometry; differential thermal anal.	crystal structure; phase tran- sitions; lattice dynamics	A. T. Horton
$C_{12}H_{18}N_2Ni_3O_{18} \cdot 8H_2O$ [$(H_2O)_3(C_2H_3N_3)_3$ $Ni]_2-Ni(NO_3)_6(H_2O)_2$		$C_2H_3N_3$ (1,2,4- triazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	optical and magnetic properties of polynuclear species	C. W. Reimann M. Zocchi, "The Structure of the Trinuclear Cation Bis-[μ -(tri-1,2,4- triazolo- N^1, N^2)- triaquonickel] nickel", Chem. Comm., 272 (1968).
$C_{12}H_{24}N_{14}NiO_6$ $(C_2H_4N_2)_6Ni(NO_3)_2$		$C_3H_4N_2$ (pyra- zole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	study of competition between hydro- gen bonding and coordinate bonding in cationic packing	C. W. Reimann, A. Santoro, and A. D. Mighell, "The Cry- stal and Molecular Structure of Hexa- pyrazolenickel- (II) Nitrate, $Ni(C_3H_4)_6WO_3 \cdot 2H_2O$ ", Acta Cryst., B26, (1970).
$C_{18}H_{24}MN_{14}O_6$ $(C_3H_4N_2)_6Cd(NO_3)_2$ $(C_3H_4N_2)_6Co(NO_3)_2$ $(C_3H_4N_2)_6Ni(NO_3)_2$	M = Cd, Co, and Ni	$C_3H_4N_2$ (imida- zole) + $M(NO_3)_2$ + H_2O	single crystal x-ray anal.	studies of single crystal visible and vibrational spectra	A. Santoro, A. D. Mighell, M. Zocchi, and C. W. Reimann, "The Crystal and Molecular Structure of Hexakis (Imida- zole) Nickel (II) Nitrate, $(C_3H_4N_3)_6$ $Ni(NO_3)_2$ ", Acta Cryst., B25, 842 (1969).

$C_{20}H_{42}$ n- $C_{20}H_{42}$ n-eicosane	2" dia. x 4"	melt solidi- fication	electrical, optical ab- sorption measurements	detr. free carrier mobility and G values; radiolysis measurements by use of optical ab- sorption techniques	A. T. Horton
CaF_2 also Gd-doped (0.1 to 1.0%)	1 x 3 x 5 mm	commercially obtained; cry- stals annealed in He + HF at 500-1200°C, rapidly quenched	absorption (200 to 2500 nm), EPR spectrometry; chemical anal. for Gd and accidental impurities	controlled point-defect equilibria studies; oxygen free	A. Franklin A. Franklin, "Mass Transport in Non- metallic Solids", Proc. of Mtg. of Basic Science Div. of British Ceramic Soc., London, Dec. 1969, in press. A. Franklin and S. Marzullo, "Orientation Kinetics of Gd^{3+} -f- Interstitial Pairs in CaF_2 ", J. Physics C, British Institute of Physics, to be published.
$CaGeO_5Ti$ $CaTiGeO_5$	1 x 1 x 2 mm	melt solidi- fication from stoichiometric composition	spectrochemical anal.; optical, x-ray diffracto- metry (powder and single crystal)	isostructural with synthetic CaO_5SiTi	C. R. Robbins C. R. Robbins "Synthetic $CaTiSiO_5$ and its Germanium Analogue ($CaTiGeO_5$)", Mat. Res. Bull., 3, 693 (1968).
$CaMoO_4$	see under $MMoO_4$				

CaO ₅ SiTi	1 x 2.5 cm (1)	melt solidification (2), and Czochralski growth (1)	spectrochemical anal.; optical, x-ray diffractometry [powder and single crystal (2)]	serves as type structure for pure CaTiSiO ₅ (space group P ₂ /n); mineral with impurities, CaTiSiO ₄ (O,OH,F), has symmetry C ₂ /c; see also CaGeO ₅ Ti	C. R. Robbins and W. S. Brower	(1) W. S. Brower and C. R. Robbins, "Growth of CaTiSiO ₅ by the Czochralski Method", J. Cryst. Growth, <u>3</u> , (1969). (2) See ref. under CaGeO ₅ Ti.
CaO ₄ W CaWO ₄	see under MO ₄ W					

Ca ₃ Fe ₂ MO ₁₂ Si ₂	M = Ti,Zr	slow cooling of Li ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	crystallographic sites detm. for both Ti and Zr in garnet structure	H. S. Peiser	J. Ito and C. Frondel, "Synthetic Zirconium and Titanium Garnets", Amer. Mineral., <u>52</u> , 773 (1967).
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Ca₃TiFe₂Si₂O₁₂
titanium garnet, dark brown, 2 mm also polycrystalline

Ca₃ZrFe₂Si₂O₁₂
zirconium garnet, chestnut brown, 0.55 mm, also polycrystalline

CdF ₂ also with dopings	1 cm	Bridgman growth	electrical		W. R. Hosler W. S. Brower	P. Eisenberger and P. S. Pershan, "Electron Spin-Resonance and Infrared Studies of Semiconducting, Rare-Earth-Doped CdF ₂ ", Phys. Rev., <u>167</u> , 292 (1968).
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ClCu	~ 1 cm	Vapor and Bridgman growth; prepd. by D. E. Roberts, also supplied by A. F. Armington AFRL, Bedford, Mass., and by A. Linz, Mass. Inst. Tech., Cambridge, Mass.	optical, dielectric	optical modulator	A. Feldman D. Horowitz	F. Sterzer, et al., "Cuprous Chloride Light Modulators", J. Opt. Soc. Am., 54, 62 (1964). A. Feldman and D. Horowitz, "Refractive Index of CuCl", J. Opt. Soc. Am. 59, 1406 (1969). NBS Tech. Rpt. 10013.
ClD ₄ N ND ₄ Cl	3 x 3 x 13 mm see also H ₂ NX	evaporation aq. soln. ND ₄ Cl + deuterourea	NMR (D, ¹⁴ N) spectrometry	measurement of quadrupole coupling const. for D on ND ₄ ⁺ configurational; torsional motion of ND ₄ ⁺ ; crystal structure	R. A. Forman M. Linzer	M. Linzer and R. A. Forman, "NMR Studies of Single Crystal ND ₄ Cl", J. Chem. Phys., 46, 4690 (1967).
ClO ₃ Na NaClO ₃	1 x 1 in.	controlled soln. growth	optical microscopy (etch pits); x-ray topography	optical, x-ray measurements	A. T. Horton	R. D. Deslattes, B. G. Sinsom, A. T. Horton, "Solution Polishing of Oriented Single Crystals", Rev. of Sci. Inst., 36, 944 (1965). R. Brooks, A. T. Horton, and J. L. Torgesen, "Occlusion of Mother Liquor in Solution-Grown Crystals", J. Cryst. Growth, 2, 279 (1968).

CLT1	~ 2 mm, very soft	Bridgman growth; prepd. by D. E. Roberts	optical	piezo-optical luminescent properties	R. Z. Bachrach and F. C. Brown. "Exciton Structure of TlCl and TlBr", Phys. Rev. Ltrs., <u>21</u> , 685 (1968).
TlCl also with dopants Cd, Pb, Nd, T					A. Feldman, "Anomalous Splitting of the Exciton Peak in the Reflectivity of TlCl and TlBr", Bull. Am. Phys. Soc., <u>14</u> , 428 (1969).
					L. H. Grabner, "Edge Fluorescence in TlBr", Bull. Am. Phys. Soc., <u>14</u> , 131 (1969).

Cl ₃ CsPb	yellow, 1 cm. dia. x 2 cm.	grown by W. S. Brower, Bridgman method	dielectric constant	measured from 4.2K through phase transition at 320K	C. K. Moller, "Structure of Perovskite-like Ce Pb Trihalides", Kgel. Danske Videnskab. Selskab, Mat. Fys. Medd., <u>32</u> , 1 (1959); also see Nature, <u>182</u> , 1436 (1958).
CsPbCl ₃					

Cl ₆ K ₂ Re	~ mm size	controlled evaporation, from soln.	ultra-sonic attenuation	second and higher order phase transitions	A. T. Horton
K ₂ ReCl ₆					

CoM ₂ O ₇ Si ₂	M = Ca, Sr; dark blue, 2 mm. Ca- and Sr cobalt akermanite; also polycrystalline analogues with M = Cd, Cu, Fe, Mg, and Mn	slow cooling, flux Na ₂ WO ₄	x-ray powder diffractometry		J. Ito and H. S. Peiser, "Distorted Tetrahedra in Strontium Copper Akermanite", J. Res. NBS, <u>73A</u> , 69 (1969).
Ca ₂ CoSi ₂ O ₇					
Sr ₂ CoSi ₂ O ₇					

$\text{CrH}_6\text{Mo}_6\text{Na}_3\text{O}_{24} \cdot \text{nH}_2\text{O}$ $\text{Na}(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot \text{nH}_2\text{O}$	$n = 8$, prepn. leads to mm size crystals, no commercial supply available	aq. soln. of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O} + \text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ at $\text{pH} = 4.5$	complete single crystal x-ray structure anal.	readily dehydrates to a composition approximating $(\text{Na}_3(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot 2\text{H}_2\text{O})$	A. Perloff (1) A. Perloff, "The Crystal Structure of Sodium Hexamolybdochromate (III) Octahydrate: $\text{Na}_3(\text{CrMo}_6\text{O}_{24}\text{H}_6) \cdot 8\text{H}_2\text{O}$ ", Inorg. Chem., in press. (2) A. Perloff, Doctoral Dissertation, Georgetown Univ. (1966). (3) G. A. Tsigdinos, Doctoral Dissertation, Boston Univ. (1961). (1) A. Perloff, paper in prepn.
$\text{CrH}_4\text{NO}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$ $\text{NH}_4\text{Cr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	$n = 13$, prepn. leads to mm size crystals, no commercial supply available	crystallizes on evaporation of aq. soln. of octahydrate at room temp.	complete single crystal x-ray structure anal.	readily dehydrates to a lower hydrate	G. A. Candela S. E. Stokowski* ferro- electric see also $\text{Al}_{n-1}\text{Cr}_n\text{--}$ $\text{KOs}_2 \cdot 12\text{H}_2\text{O}$
$\text{CuO}_4\text{S} \cdot 5\text{H}_2\text{O}$ $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	1 cm^2	controlled evaporation from soln.	NMR spectrometry	NMR spectroscopy, wave guide experiments	A. T. Horton

Cu ₂ O	1.5 x 6 cm	grown from melt	optical and electrical measurements; air sensitive	W. S. Brower H. S. Parker	W. S. Brower and H. S. Parker, "Melt Growth of Large Cuprous Oxide Single Crystals", Abstracts, ACGC- NBS Conf. on Crystal Growth, August 11-13, 1969, Washington, D. C., p. 115.
		prepd. by W. S. Brower	electrical	R. A. Forman W. R. Hosler K. F. Young	oxide- semiconductor dielectric constant
F ₂ MgNa ₂ O ₂₄ Si ₆	largest crystal 2 x 2 x 6 cm colorless purple green yellow	flux (NaF) isothermal evaporation	x-ray powder diffraction anal.; visible emission spectrometry	H. S. Peiser	J. Ito, "Silicate Apatites and Oxya- patites", Amer. Mineral., 53, 890 (1968).
M = Gd La Nd Pr Sm					
F ₂ Mg MgF ₂	~ 1 cm ³	pulled from melt, D. E. Roberts	optical; EPR spectrometry	M. I. Cohen	R. F. Blunt and M. I. Cohen, "Irradiation- Induced Color Centers in Magnesium Fluoride", Phys. Rev., 153, 1031 1967).
F ₂ Pb PbF ₂ also Y, rare- earth dopings		Bridgeman growth		W. R. Hosler W. S. Brower	D. A. Jones, "Growth of Lead Fluoride Crystals from the Melt", Proc. Phys. Soc., 68B, 165 (1955).

H ₂ O - VII ice VII	structure VII	from liquid at 100°C and 30 Kb	x-ray diffractometry	unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weir*	C. E. Weir, S. Block, and G. Piermarini, "Single Crystal X-ray Diffraction at High Pressures", J. Res. NBS, 69C, 275 (1965).
H ₄ NX NH ₄ X X = Br, Cl, I also with Tl doping	~ 1 cm ³	from aq. soln. by evaporation	optical	relation to alkali halides	R. A. Forman	
H ₆ NO ₄ P NH ₄ H ₂ PO ₄ "ADP"	3/4 x 3 in	controlled growth from soln.	optical microscopy (etch pits); x-ray topography	monochrometers, electro-optics, x-ray reference crystals	A. T. Horton	R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Preliminary Studies on the Characterization of Solution-Grown ADP Crystals", Adv. in X-ray Anal., 8, p. 315 (1965). R. D. Deslattes, J. L. Torgesen, B. Paretzkin, and A. T. Horton, "Observation of Dislocations in ADP: Production of Dislocation-Free Crystals", J. Appl. Phys., 37, 541 (1966).
InNaO ₆ Si ₂ NaInSi ₂ O ₆	colorless indium aegirine, 1 cm; also obtained as polycrystalline	slow cooling from Na ₂ WO ₄ or Na ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	see also NaO ₆ ScSi ₂	H. S. Peiser	J. Ito, "Synthetic Indium Silicates and Hydrogarnet", Am. Mineral., 53, 1663 (1968).

See ref. under Br₂.

S. Block
G. Piermarini
C. E. Weir*

detm. unit
cell, space
group, phase
equilibrium

x-ray diffractometry

obtained from
KNO₃ II at
elevated temp.
and pressure

structure
III

KNO₃ III

from saturated aq.
soln. KNO₃
at 100°C,
20 Kb

structure
IV

KNO₃ IV

M. I. Cohen, et al.,
"Lattice Absorption
in Strontium Titanate", Bull. Am.
Phys. Soc., 14,
498 (1969).

M. I. Cohen
W. R. Hosler

oxide
semiconductor

electrical, optical

prepd. by
W. S. Brower;
also obtained
with doping
from A.
Linz, Mass.
Inst. Tech.,
Cambridge,
Mass.; flux
growth

KO₃Ta
KTaO₃, also
doped with
Mn

W. R. Hosler and H.
P. R. Frederikse,
"Magnetoresistive
Effects in KTaO₃",
Sol. State Comm.,
7, 1443 (1969).

MnMoO ₄	M = Ca, Pb, Sr			see also MnMoO ₄		W. S. Brower and P. H. Fang, "Dielectric Constants of PbMoO ₄ and CaMoO ₄ ", Phys. Rev., <u>149</u> , 646 (1966).
CaMoO ₄ PbMoO ₄ SrMoO ₄	1 x 3 cm	melt grown	x-ray topography; dielectric constant, etch pits	acousto-optic devices	W. S. Brower	W. S. Brower and P. H. Fang, "Dielectric Constants of PbWO ₄ and CaWO ₄ ", J. Appl. Phys., <u>38</u> , 2391 (1967).
			mechanical	elastic constants of CaMoO ₄	J. Wachtman, Jr.	J. B. Wachtman, Jr., W. S. Brower, Jr. and E. N. Farabaugh, "Elastic Constants of Single Crystal Calcium Molybdate (CaMoO ₄)", J. Am. Ceram. Soc., <u>51</u> , 341 (1968).
MO ₄ W	M = Ca, Pb, Sr, Zn; 1 x 3 cm	melt grown	x-ray topography; dielectric constant; etch pits	acousto-optical devices	W. S. Brower	W. A. Bonner, "Growth of Large Single Crystal Lead Molybdate for Acousto-Optic Applications", Abstr. ACG-NBS Conf., August, 1969, p. 116.
CaWO ₄ PbWO ₄ SrWO ₄ ZnWO ₄						
ZnWO ₄	1 cm		optical	magneto-optical properties	R. F. Blunt	

MnMoO ₄	1 cm	pulled from melt; prepd. by D. E. Roberts	optical	magneto-optical properties	R. F. Blunt	See also under MMoO ₄ and MO ₄ W for additional refs. to Scheelite crystals.
MnO ₄ W MnWO ₄	1 cm	pulled from melt; prepd. by D. E. Roberts.	optical	magneto-optical properties	R. F. Blunt	See also MO ₄ W.
MoO ₄ Pb MoO ₄ Sr	see MMoO ₄					
NaO ₆ ScSi ₂ NaScSi ₂ O ₆	colorless, scandium aegirine, 0.5 cm; also as poly-crystalline	slow cooling from Na ₂ WO ₄ or Na ₂ MoO ₄ flux	powder and precession x-ray diffraction anal.	see also InNaO ₆ Si ₂	H. S. Peiser	J. Ito and C. Frondel, "Synthesis of Scandium Analogues of Aegirine, Andradite and Melanotekite", Am. Mineral., <u>53</u> , 1276 (1968).
NiTi TiNi	wire	obtained from NOL, White Oak, Md.	electrical and magnetic	exhibits martensitic transition; i.e., shows mechanical memory through transition	W. R. Hosler	F. E. Wang, B. F. DeSavage, W. J. Buehler, and W. R. Hosler, "The Irreversible Critical Range in the TiNi Transition", J. Appl. Phys. <u>39</u> , 2166 (1968).
O ₂ Th ThO ₂	2 x 1 x 1 cm	selected from arc melt by G. R. Findlay, Norton Co.	Laue diffraction	elastic constants	J. Wachtman, Jr.	P. B. Macedo, W. Capps, and J. B. Wachtman, Jr., "Elastic Constants of Single Crystal ThO ₂ at 25°C", J. Am. Ceram. Soc., <u>47</u> , 651 (1964).

O ₂ Ti	single crystal bar	Verneuil process, reheated in O ₂	compressibility to 10 Kb	C. E. Weir*	See ref. under Al ₂ O ₃ .
TiO ₂	1 cm	flame fusion, prepd. by D. E. Roberts	EPR spectrometry; electrical, optical	R. F. Blunt	T. Chang, "Electron Spin Resonance of Mo ³⁺ in Rutile", Phys. Rev., <u>136</u> , A413 (1964); "Paramagnetic Resonance Spectrum of W ⁵⁺ in Rutile (TiO ₂)", Phys. Rev., <u>147</u> , 264 (1966).
TiO ₂ pure, also with dopants: Al, Cr, Cu, Fe, Mo, Nb, rare earths, V, W, Zn				T. Chang Mo ³⁺ in Rutile", Phys. Rev., <u>136</u> , A413 (1964); "Paramagnetic Resonance Spectrum of W ⁵⁺ in Rutile (TiO ₂)", Phys. Rev., <u>147</u> , 264 (1966).	
				H. P. R. Frederikse, "Recent Studies on Rutile", J. Appl. Phys. Suppl., <u>32</u> , 2211 (1961).	
TiO ₂ pure, also with dopants: Cr, Ni; oxidized and reduced conditions	single crystal rod	flame fusion	Laue diffraction patterns	J. Wachtman, Jr.	J. B. Wachtman, Jr., W. E. Tefft, and D. G. Lam, Jr., "Elastic Constants of Rutile (TiO ₂)", J. Res. NBS, <u>66A</u> , 465 (1962).
			elastic constants; internal friction	S. Spinner and J. B. Wachtman, Jr., "Some Elastic Compliances of Single Crystal Rutile from 25 to 1000°C", J. Res. NBS, <u>68A</u> , 669 (1964).	
				J. B. Wachtman, Jr. and L. R. Doyle, "Internal Friction in Rutile Containing Point Defects", Phys. Rev., <u>135</u> , A276 (1964).	
				J. B. Wachtman, Jr., S. Spinner, W. S. Brower, T. Fridinger, and R. W. Dickson, "Internal Friction in Rutile Containing Ni or Cr", Phys. Rev., <u>148</u> , 811 (1966).	

O ₂ U	3 x 1 x 1 cm	selected from arc melt by H. J. Anderson of Hanford Atomic Products Operation	density, coulometric titration, spec. analysis, Laue diffraction	elastic constants	J. Wachtman, Jr. J. B. Wachtman, Jr., M. L. Wheat, H. J. Anderson and J. L. Bates, "Elastic Constants of Single Crystal UO ₂ at 25°C", J. Nuclear Materials, <u>16</u> , 39 (1965).
O ₃ PbSi	alamosite, colorless, 2 mm; also prepd. as polycrystal- line	hydrothermal, spontaneous nucleation isothermal	x-ray structural anal. (M. Mydlar, Ceramic Engr. Dept., Univ. of Mo.)	structure	J. Ito, "Synthesis of Some Lead Calcium Zinc Silicates", Amer. Mineral., <u>53</u> , 231 (1968).
PbSiO ₃					

O_3SrTi	1 cm	flame fusion; also supplied by D. Beals, National Lead Co., South Amboy, N. J.	optical, electrical; EPR spectrometry	insulator; semiconductor, super- conductor; pseudo- ferroelectric; photochromic	T. Chang M. I. Cohen A. Feldman L. H. Grabner W. R. Hosler A. H. Kahn	A. H. Kahn and A. J. Leyendecker, "Electronic Energy Bands in Strontium Titanate", Phys. Rev., <u>135</u> , A1321 (1964).
$SrTiO_3$ pure, also with dopants: Al, Ga, Nb						H. P. R. Frederikse, W. R. Hosler, and W. R. Thurber, "Experi- mental Evidence Concerning the Conduction and Band of $SrTiO_3$ ", J. Phys. Soc. Japan, 21, Suppl., 32 (1966).
						J. F. Schooley and W. R. Thurber, "Super- conductivity in Semiconducting $SrTiO_3$ ", J. Phys. Soc. Japan, 21, Suppl., 639 (1966).
						A. Feldman and D. Horowitz, "Stress- Induced Dichroism at the Absorption Edge of Strontium Titanate", Sol. State Comm., <u>6</u> , 607 (1968).
$SrTiO_3$	1 - 2.5 mm	melt solidi- fication using SiO_2 as flux	spectrochemical, optical and x-ray powder diffrac- tion	prepd. in absence of fluorides, etc.	C. R. Robbins	C. R. Robbins, "Growth of Strontium Titanate from a Silica Flux", J. Cryst. Growth, <u>2</u> , 402 (1968).

V. POLYCRYSTALS

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION		REFERENCES (Author, Title, Journal)
				COMMENTS		
Ag	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials, SRM # 748	Knudsen, torque Knudsen, and mass spectrometric detm. of vapor pressure as function of temperature; detm. heat of sub- limation	calibrn. of vapor pressure apparatus and techniques (10-8 to 10-3 atm., 1000-1600°K); this and other elements (see Au, Cd, Pt, and W) will each cover a different tem- perature range; these five mat- erials cover 400-3000°C	R. C. Paule Mandel, NBS Special Publication # 260.	R. C. Paule and J. Mandel, NBS Special Publication # 260.
AgNO ₃ "high-AgNO ₃ "	polycrystal- line surface, α~50μ	high temp- erature x-ray diffracto- meter furnace	x-ray powder diffractometry	vapor pressure, Knudsen effusion collection	E. R. Plante	See ref. above.
AlN aluminum nitride	100-300 mesh	four commer- cial sources	x-ray diffractometry	unstable at room temp.	E. M. Levin	E. M. Levin, "X-Ray Determination of the Thermal Expansion of Silver Nitrate", J. Am. Ceram. Soc., <u>52</u> , 53 (1969).
Al ₂ BaO ₄ BaAl ₂ O ₄	γ-Al ₂ O ₃ + BaCO ₃ at 1200°C for 1.5 hr.	x-ray diffractometry; petrographic microscopy	parent com- pound for study of transport chemistry		F. E. Brinckman	H. Swanson et al., "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 5, p. 11 (1967).

Al ₂ O ₃	See also IV. SINGLE CRYSTALS	(a) commercial powder and crystals (b) powder purified by solar furnace	melting point detm.	melting point standard; Reference Material	S. J. Schneider and C. L. McDaniel, "Effect of Environment Upon the Melting Point of Al ₂ O ₃ ", J. Res. NBS, <u>71A</u> , 317 (1967).
					S. J. Schneider, "Cooperative Determination of the Melting Point of Al ₂ O ₃ ", to be published in Pure and Applied Chemistry (1970).
Au	high purity wire; 0.055 in. dia. x 6 in; Standard Reference Material # 745	special prepn. for Office of Standard Reference Materials, SRM # 745	see under Ag	temp. range 1300-2100°K; see also under Ag	R. C. Paule and J. Mandel, "Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold", NBS Special Publication # 260- 19.
	0.055" wire	available as SRM # 745	see under Ag	see under Ag	See ref. above.
BF ₄ M KBF ₄ NaBF ₄	M = K, Na; obsd. also in aq. soln.	commercial	NMR (¹¹ B, ¹⁹ F) spectrometry	model compounds for high resolution and broad- line NMR; J _{B-F} as related to solvents	F. E. Brinckman T. C. Farrar
BH ₃ O ₃ H ₃ BO ₃		commercial	compressibility to 10 Kb	see also Al ₂ O ₃ under Section IV.	C. E. Weir, "Compress- ibility of Eleven In- organic Materials", J. Res. NBS, <u>69A</u> , 29 (1965).

BH ₄ M	M = Li, K, Na; obsd. also in aq. soln.	commercial	NMR (¹ H, ¹¹ B) spectrometry	model com- pounds, detm. phase trans- itions and motions in solids	T. C. Farrar	T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, 73A, 195 (1969).
B ₂ O ₃ Sc ScB ₂ O ₃	~50μ part- icles, calcite type	solid state rxn. of Sc ₂ O ₃ + B ₂ O ₃ in sealed tube	x-ray powder diffractometry, polarizing microscopy	phase diagram	E. M. Levin	E. M. Levin, "System Sc ₂ O ₃ -B ₂ O ₃ ", J. Am. Ceram. Soc., 50, 53 (1967).
B ₂ O ₃		controlled dehydration of H ₃ B ₃ O ₃	compressibility to 10 Kb		C. E. Weir*	See ref. under BH ₃ O ₃ .
B ₂ O ₃ •3Nb ₂ O ₅ "3Nb ₂ O ₅ •B ₂ O ₃ "	~50μ	solid state rxn. of B ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube	x-ray powder diffractometry	phase diagram	E. M. Levin	E. M. Levin, "Phase Equilibria on the System Niobium Pentoxide-Boric Oxide", J. Res. NBS, 70A, 11 (1966).
Borate minerals, anhydrous	~80 in number, fine powders	metal oxide + H ₃ B ₃ O ₃ at elevated temp.	infrared (2000- 300 cm ⁻¹) spectro- metry	correlation of anion and spectra	C. E. Weir*	C. E. Weir and R. A. Schroeder, "Infrared Spectra of the Crystalline Inorganic Borates", J. Res. NBS, 68A, 465 (1965).
Borate minerals, hydrated	~42 natural and synthetic	obtained from the Smithsonian Inst. U. S. Geological Survey, Wash- ington, D. C. and Menlo Park, Calif.	infrared spectro- metry, principally in 2000-300 cm ⁻¹ region	evaluation of limita- tions of spectra in identification, effects of treatment on spectra	C. E. Weir*	C. E. Weir, "Infrared Spectra of the Hydrated Borates", J. Res. NBS, 70A, 153 (1966).
BaF ₃ Li LiBaF ₃		fuse stoichio- metric amt. LiF + BaF ₂ at 800°, annealed at 500° to re- move BaF ₂ ppt.	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 35.

BaGe ₃ O ₉ Ti	20-40μ	solid state rxn. of BaTiO ₃ + GeO ₂ in sealed Pt tube at 1160°C	optical and x-ray powder diffractometry	stable from 1132° to 1235°C but readily obtain- able by quench- ing to ambient temp.; serves as a structural intermediate between BaGe ₄ O ₉ and BaTiSi ₃ O ₉	C. R. Robbins	C. R. Robbins, "The Compound BaTiGe ₃ O ₉ ", J. Am. Ceram. Soc., <u>43</u> , 610 (1960).
BaTiGe ₃ O ₉						
Ba ₂ CuO ₁₀ Si ₂ Y ₂	polycrystal- line for all possible combinations with MLn ₂ Be ₂ - Si ₂ O ₁₀ , where M=Mg, Ni, Zn, Co, Cu, Fe, Mn, and Cd; single crystal (lmm) also studied	slow cooling of solute in Na ₂ WO ₄ or Na ₂ WO ₇ flux	precession and powder x-ray diffractometry		H. S. Peiser	J. Ito, "The Synthesis of Gadolinite", Proc. Jap. Acad., <u>41</u> , 404 (1965).
CuY ₂ Be ₂ Si ₂ O ₁₀						J. Ito, "A Note on the Gadolinite Synthesis", Proc. J. Acad., <u>42</u> , 634 (1966).
						J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., <u>52</u> , 1523 (1967).
BiNbO ₄	see also: BiO ₄ Ta BiO ₄ V NbO ₄ Sb O ₄ SbTa O ₄ SbV	solid state rxn. of Bi ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tubes at 950 - 1250°C	x-ray powder diffractometry		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Synthesis and Stability of Bismutotantalite, Stibiotantalite and Chemically Similar ABO ₄ Compounds", Am. Mineral., <u>48</u> , 1348 (1963).
BiO ₄ Ta BiTaO ₄		solid state rxn. of Bi ₂ O ₃ + Ta ₂ O ₅ in sealed Pt tubes at 845 - 1150°C	see BiNbO ₄		R. S. Roth J. L. Waring	See ref. under BiNbO ₄ .
BiO ₄ V BiVO ₄		solid state rxn. of Bi ₂ O ₃ + V ₂ O ₅ in sealed Pt tubes at 500-1000°C	see BiNbO ₄		R. S. Roth J. L. Waring	See ref. under BiNbO ₄ .

Bi₂O₃ E. M. Levin and R. S. Roth, "Polymorphism of Bismuth Sesquioxide. I. Pure Bi₂O₃", J. Res. NBS, 68A, 189 (1964); Ibid., II, 197-206.

obsd. at room temp. as metastable phase

differential thermal anal.; x-ray diffractometry

high-temp. x-ray diffractometer furnace; programmed heating and cooling

cubic

tetragonal

Br₂O₂Pb₃

Pb₃O₂Br₂

heat stoichiometric PbO (red) + PbBr₂ in Au boat, at 350°C for 45 min.

x-ray diffractometry, petrographic microscopy

H. Swanson J. deGroot H. McMurdie See ref. under Al₂BaO₄, p. 32.

Br₃Ti

TiBr₃

dark purple, fine powder or small crystals; see also Cl₃Ti

TiBr₄+H₂ → using hot Ir filament

elemental anal. for Ti, Br

R. B. Johannesen

sublimes at ~ 500°C with disproportionation, strong reducing agent; catalyst for olefin polymerization; air/moisture sensitive

J. M. Sherfey, "Titanium-(III) Chloride and Titanium (III) Bromide", Inorg. Synth., 6, 57 (1960).

Br₄Ti

TiBr₄

yellow, m.p. 38°C

TiCl₄+HBr → at b.p. of TiCl₄-n Br_n

elemental anal., m.p., b.p.

R. B. Johannesen

strong Lewis Acid properties, air/moisture sensitive

R. B. Johannesen and G. L. Gordon, "Titanium (IV) Bromide", Inorg. Synth., 9, 46 (1967).

Br₆H₈N₂Os

(NH₄)₂OsBr₆

mixed crystals (solid soln.) of (NH₄)₂-(Os-Pt)Br₆, also prepd. where Os/Os +Pt = 0.0763

(a) OsO₄ + HBr(aq) (b) (a) + NH₄Br filament

elemental anal., x-ray diffractometry

R. B. Johannesen

parent compounds for magnetic susceptibility studies

R. B. Johannesen and G. A. Candela, "Magnetic Susceptibilities and Dilution Effects in Low-Spin d⁴ Complexes: Osmium (IV)", Inorg. Chem., 2, 67 (1963).

CSi	cubic, ~200 mesh	Si + C above m.p. of Si	x-ray diffractometry, vapor pressure	other polymorphs present, C-contaminated	E. R. Plante
SiC					
C ₂ Na ₂ O ₄ Na ₂ C ₂ O ₄ sodium oxalate	NBS standard sample	NBS standard sample No. 40d	assay 99.9% Na ₂ C ₂ O ₄ ; x-ray diffractometry, petrographic microscopy	H. Swanson	H. Swanson, et al, "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 6, p. 70 (1968).
C ₃ Al ₄	large crystallites embedded in polycrystalline matrix	Al(l) + C at 1400°C	x-ray diffractometry, vapor pressure	usually contains excess C; moisture sensitive	E. R. Plante and C. H. Schreyer, "Dissociation Pressure of Aluminum Carbide Using a Rotating Knudsen Cell", J. Res. NBS, 70A, 253 (1966).
C ₄ HCoO ₄ HCo(CO) ₄	unstable liquid, studied as solid	see SECTION II	see SECTION II	see SECTION II	See SECTION II.
C ₄ H ₁₂ F ₂ O ₄ W (CH ₃ O) ₄ WF ₂	colorless crystals m.p. 81-83°C cis-stereoisomer only	WF ₆ + 4CH ₃ OSi(CH ₃) ₃ in C ₆ F ₆	NMR(¹ H, ¹⁹ F) mol. wt. elemental analysis	one of a series of stable tungsten (VI) compounds; easily purified by sublimation; <u>cis</u> -difluoro configuration; provides basis for W (VI) stereochemistry; air/moisture sensitive.	F. E. Brinckman L. B. Handy* F. E. Brinckman, F. E. Brinckman, "Chemistry of the Methoxyfluorotungsten (VI) Series", Chem. Comm., 214 (1970).

$C_6H_{10}CuO_4 \cdot H_2O$ Copper (II) propionate monohydrate	also Zn- doped	heat $Cu(Zn)CO_3$ + $[CH_3CH_2CO]_2O$ + H_2O	EPR spectrometry	model com- pounds for detm. magnetic parameters in dimeric $Cu(II)$ alkanoates from EPR studies of polycrystalline samples	M. Linzer G. F. Kokoszka, M. Linzer, G. Gordon, "Electron Paramagnetic Resonance Spectra of Polycrystalline Dimeric Complexes. Copper Propionate Monohydrate and Zinc- Doped Copper Pro- pionate Monohydrate", Inorg. Chem., <u>7</u> , 1730 (1968).	G. F. Kokoszka, M. Linzer, G. Gordon, "Electron Paramagnetic Resonance Spectra of Polycrystalline Dimeric Complexes. Copper Propionate Monohydrate and Zinc- Doped Copper Pro- pionate Monohydrate", Inorg. Chem., <u>7</u> , 1730 (1968).
$C_6H_{18}HgSi_2$ $[(CH_3)_3Si]_2Hg$	yellow	$(CH_3)_3SiX +$ Na/Hg at 55°C (X=Cl, Br) (1)	NMR (1H) spectrometry	source of trimethyl- silyl group or radical (2); toxic, volatile Hg compound, soln. light- sensitive	F. E. Brinckman L. B. Handy# (1) E. Wiberg, et. al, "Recent Developments in the Chemistry of Metal Silyls of the Type $M(SiR_3)_n$ ", Angew. Chem. internat. Edit., <u>2</u> , 507 (1963). (2) C. Eaborn, R. A. Jackson, and R. W. Walsingham, "The Reduction of Organo- silicon Radicals in Solution", Chem. Comm., <u>1965</u> , 300.	(1) E. Wiberg, et. al, "Recent Developments in the Chemistry of Metal Silyls of the Type $M(SiR_3)_n$ ", Angew. Chem. internat. Edit., <u>2</u> , 507 (1963). (2) C. Eaborn, R. A. Jackson, and R. W. Walsingham, "The Reduction of Organo- silicon Radicals in Solution", Chem. Comm., <u>1965</u> , 300.
$C_{12}H_{36}LiN_2NaSi_4$ $[(CH_3)_3Si]_2N < \begin{smallmatrix} Li \\ Na \end{smallmatrix}$ > $N[Si(CH_3)_3]_2$		metallation of N-H on disilazanes; sample supplied by Prof. U. Wannagat, T. U. Carolo-Wilhelmina, Braunschweig, Germany	see ref.?, NMR (1H , 7Li , ^{14}N , ^{23}Na) spectrometry	first ex- ample of NMR measure- ment of a mixed dimeric electron- deficient com- pound in the solid state	M. Linzer D. Mootz, A. Zinnius, and B. Bottcher, "Association in the Solid State of Bis(trimethylsilyl) amidolithium and Methyltrimethyl- silylanatoberyllium", Angew. Chem., <u>81</u> , 398 (1969).	D. Mootz, A. Zinnius, and B. Bottcher, "Association in the Solid State of Bis(trimethylsilyl) amidolithium and Methyltrimethyl- silylanatoberyllium", Angew. Chem., <u>81</u> , 398 (1969).
$C_{20}H_{16}Cu_2N_{20} \cdot 4H_2O$ $Cu_2(C_5H_4N_5)_4 \cdot$ $4H_2O$ $C_{20}H_{20}Cl_{14}Cu_2N_{20}O_{16}$ $\cdot 3H_2O$ $Cu_2(C_5H_5N_5)_4 \cdot$ $(ClO_4)_4 \cdot 3H_2O$	$(C_5H_5N) =$ adenine moiety	$Cu(ClO_4)_2(aq)$ + adenine, adjust pH	chemical anal., optical and EPR spectroscopy	metal-metal bond inter- actions pertinent to biochemical processes involving nucleic acids	G. F. Kokoszka R. W. Duerst, S. J. Baum, and G. F. Kokoszka, "Exchange Coupling in Two Dimeric Copper Adenine Complexes", Nature, <u>222</u> , 665 (1969).	R. W. Duerst, S. J. Baum, and G. F. Kokoszka, "Exchange Coupling in Two Dimeric Copper Adenine Complexes", Nature, <u>222</u> , 665 (1969).

CaCl ₃ Cs CsCaCl ₃	CsCl + CaCl ₂ at ~ 900°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 21.	
CaNa ₂ O ₈ S ₂ Na ₂ Ca(SO ₄) ₂	CaCl ₂ +Na ₂ SO ₄ in aq. soln.; intermediate CaSO ₄ forms, desired product after several hr., washed with alcohol	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 59.	
Cd	high purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials; SRM # 746	see Ag	temp. range 400-600°K; see also under Ag	See ref. under Ag.
CdCl ₃ Cs CsCdCl ₃	stoichiometric CsCl+CdCl ₂ fusion	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 19.	
CdCl ₃ H ₄ N NH ₄ CdCl ₃	stoichiometric NH ₄ Cl+CdCl ₂ (aq. soln.) → ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 6.	
CdCl ₃ K KCdCl ₃	fuse KCl + CdCl ₂ ·2.5H ₂ O at 550°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 38.	
CdCl ₃ Rb	orthorhombic	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 41.	
	tetragonal	fuse RbCl + CdCl ₂ (anhydr.) at ~ 500°C		Same, p. 43.	

CdCr ₂ O ₄	(a) CdO + Cr ₂ O ₃ heated in vacuo at 300°C for 17 hr. (b) ground, pelletized, heated at 1050°C for 1.5 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 16.
CdO ₄ S·H ₂ O CdSO ₄ ·H ₂ O	(a) crystallized from aq. soln. at 95°C; (b) also prepd. from CdSO ₄ or CdSO ₄ ·8H ₂ O on prolonged exposure to air of RH = 50%.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 10.
3CdO ₄ S·8H ₂ O	obtained from Johnson, Matthey & Co., Ltd.	high humidity necessary to prevent formation of monohydrate		same, p. 8.
ClH ₄ NO NH ₃ OHCl	obtained from Naval Ordnance Plant, Indian Head, Md.	NMR (1H) spectrometry, vapor pressure	T. C. Farrar	
Cl ₂ P PCl ₂ (free radical)	at 77K in PCl ₃ matrix, C _{2v} symmetry	EPR spectrometry	F. E. Brinckman G. F. Kokoszka	G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", Chem. Comm., 349 (1969).
Cl ₄ P PCl ₄ (free radical)	C _{2v} symmetry	formation of PCl ₄ by sequential rxns.; evaluation of molecular bonding, relation to chlorophosphorylation via radical intermediates	G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", J. Am. Chem. Soc., 92, 1199 (1970).	

Cl ₃ CoCs CsCoCl ₃	co-pptd. CsCl + CoCl ₂ heated in sealed glass tube at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 11.
Cl ₃ CoH ₄ N NH ₄ CoCl ₃	co-pptd. NH ₄ Cl + CoCl ₂ heated in sealed glass tube at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 5.
Cl ₃ CoRb RbCoCl ₃	co-pptd. RbCl + CoCl ₂ heated in sealed glass tube at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 57.
Cl ₃ CsCu CsCuCl ₃	dark red hex. prisms terminated by bipyramids crystallized from aq. CsCl + CuCl ₂	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 22.
Cl ₃ CsNi CsNiCl ₃	co-pptd. CsCl + NiCl ₂ heated in sealed glass tube at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 12.
Cl ₃ CsPb CsPbCl ₃	fuse CsCl + PbCl ₂ at ~ 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 24. See also in SECTION IV.
Cl ₃ CsSr CsSrCl ₃	SrCl ₂ + CsCl melted at 900°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 13.
Cl ₃ H ₄ HgN NH ₄ HgCl ₃	pptd. from soln. of NH ₄ Cl + HgCl ₂	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 9.

Cl ₃ H ₄ NNi NH ₄ NiCl ₃	(a) NiCl ₂ · 2H ₂ O + HCl at 150°C + NiCl ₂ (b) (a) + NH ₄ Cl in sealed glass tube at 300°C for 72 hr.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 6.
Cl ₃ HgNa·2H ₂ O NaHgCl ₃ ·2H ₂ O	crystalli- zation from aq. soln. of equimolar amts of NaCl + HgCl ₂	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 66.
Cl ₃ NiRb RbNiCl ₃	co-pptd. RbCl + NiCl ₂ heated in sealed glass tube at 500°C	moderately hygroscopic	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 58.
Cl ₄ P	see Cl ₂ P			see Cl ₂ P
CoF ₃ K KCoF ₃	pptd. from mixt. Kf + CoF ₂ soln., washed	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 37.
CoNa ₂ O ₈ S ₂ ·4H ₂ O Na ₂ Co(SO ₄) ₂ ·4H ₂ O	crystallized from aq. soln. Na ₂ SO ₄ + CaSO ₄ at room temp.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 61.
Co ₂ Sb ₂ CoSb ₂ O ₆	CoC ₂ O ₄ + Sb ₂ O ₅ + at 1000°C for 30 min. in Au boat	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 26.
Co ₂ K ₂ O ₁₂ S ₃ K ₂ Co ₂ (SO ₄) ₃	K ₂ SO ₄ + CoSO ₄ melted at 600°C	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 35.

CuF ₃ K KCuF ₃	add CuCl ₂ to excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 38.
Cu ₂ O ₆ Sb ₂ CuSb ₂ O ₆	solid state rxn. of CuO + Sb ₂ O ₅ in Ag boat at 945°C in vacuo for 2 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 27.
ErO ₄ V ErVO ₄	Er ₂ O ₄ + V ₂ O ₅ heated at 1100°C for 30 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 29.
FMPO ₃ BaPO ₃ F CaPO ₃ F K ₂ PO ₃ F Na ₂ PO ₃ F	also studied in soln. Na ₂ PO ₃ F + MCl ₂ ; M= Ba,Ca,K	first detm. of absolute sign of a spin-coupling constant; high resolution, pulsed and wide-line NMR x-ray diffractometry, NMR (19F, 31P) spectrometry	T. C. Farrar A. Perloff D. VanderHart	D. L. VanderHart, H. S. Gutowsky, and T. C. Farrar, "NMR Study of BaPO ₃ : 31P and 19F Chemical- Shift Anisotropies and the Absolute Sign of the F-P Coupling Constant", J. Chem. Phys., <u>50</u> , 1050 (1969).
F ₃ FeK KFeF ₃	aq. soln. FeCl ₂ + KF → ppt., washed, heated to 400°C in vacuo	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 39.
F ₃ KMg KMgF ₃	HF + slurry of K ₂ CO ₃ + MgCO ₃ , evapo- rated to dryness; x-ray pattern sharpened by heating sample to m.p.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 42.

F ₃ KMn KMnF ₃	aq. MnCl ₂ + excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 45.
F ₃ MnNa NaMnF ₃	aq. MnCl ₂ + excess KF in soln. → ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 65.
F ₃ NaZn NaZnF ₃	soln. ZnCl ₂ + conc. soln. NaF → ppt., washed, annealed at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 74.
FeNbO ₄	solid state rxn. of Fe ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube at 1000- 1475°C	x-ray powder diffractometry	R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Ixiolite and Other Polymorphic Types of FeNbO ₄ ", Am. Mineral., 49, 242 (1964).
GdO ₄ V GdVO ₄	Gd ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ at 800°C for 15 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 30.
GeO ₂ •9Nb ₂ O ₅	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	E. M. Levin	E. M. Levin, "Phase Equilibria in the System Niobium Pentoxide-Germanium Dioxide", J. Res. NBS, 70A, 5 (1966).
Ge ₄ O ₉ Pb PbGe ₄ O ₉	20-40 μ see BaGe ₄ O ₉ under SECTION IV + GeO ₂ in sealed Pt tube at 700°C	optical and x-ray powder diffractometry	C. R. Robbins isostructural with BaGe ₄ O ₉ SrGe ₄ O ₉	C. R. Robbins and E. M. Levin, "Tetra- germanates of Strontium Lead and Barium of Formula Type AB ₄ O ₉ ", J. Res. NBS, 65A, 127 (1961).

Ge ₄ O ₉ Sr SrGe ₄ O ₉	20-40 μ	solid state rxn. of SrO + GeO ₂ in sealed Pt tube at 1200°C	optical and x-ray powder diffractometry	isostructural with BaGe ₄ O ₉ PbGe ₄ O ₉	C. R. Robbins	See ref. under Ge ₄ O ₉ Pb.
H ₂ M ₁₀ O ₂₆ P ₆ Ba ₁₀ P ₆ O ₂₄ (OH) ₂ Pb ₁₀ P ₆ O ₂₄ (OH) ₂ Sr ₁₀ P ₆ O ₂₄ (OH) ₂	1-25 μ M = Ba, Pb, Sr	solid state rxn. of BaO, PbO, SrO + P ₂ O ₅ in air at elevated temp.	x-ray diffractometry; infrared, broad-line NMR spectrometry; gravimetric anal.	illustrate nature of non-stoichiometric phases	T. Negas R. S. Roth	T. Negas and R. S. Roth, "High Temperature Dehydroxylation of Apatitic Phosphates", J. Res. NBS, 72A, 783 (1968).
H ₄ PI PH ₄ I		PH ₃ + HI \rightarrow	infrared, NMR (¹ H, ³¹ P) spectrometry, vapor pressure	phase transitions and molecular motions in solids; toxic, air/moisture sensitive	T. C. Farrar	T. C. Farrar and J. J. Rush, "Nuclear Magnetic Resonance and Neutron-Scattering Studies of Molecular Motions in Phosphonium Iodide", Proc. Second IMR Symp. (Molecular Dynamics and Structure of Solids), NBS Special Publ. 301 (1969), p. 245.
H ₄ PX PH ₄ Br PH ₄ Cl PH ₄ I	X=Br, Cl, I; volatile solid	PH ₃ + HX \rightarrow	NMR (¹ H, ³¹ P), laser Raman spectrometry; vapor pressure, neutron inelastic scattering	model compounds, phase transitions and motions in solids	T. C. Farrar	T. Tsang, T. C. Farrar, and J. J. Rush, "Proton Magnetic Resonance and Hindered Rotation in Phosphonium Halides and Ammonium Iodide", J. Chem. Phys., 49, 4403 (1968).
					J. J. Rush, A. J. Melveger, T. C. Farrar and T. Tsang, "Laser-Raman Spectra and Hindered Rotation in the Phosphonium Halides", Chem. Phys. Letters, 2, 621 (1968).	

H_9K_2Re K_2ReH_9	supplied by A. P. Ginsberg, Bell Tele- phone Labs., Murray Hill, N. J.	neutron, x-ray diffractometry, NMR (1H) spectro- metry; elemental anal.	study of molecular dynamics of two crystal- lographically different anions; illustrates different reorientation energies	T. C. Farrar R. B. Johannesen	T. C. Farrar, T. Tsang, and R. B. Johannesen, "Internal Reorientation in K_2ReH_9 via Wide- Line and Pulsed Proton Magnetic Resonance Studies", J. Chem. Phys., 51, 3595 (1969).
IrO ₂	powder oxidation of Ir by heat treat- ment in air and/or O ₂	x-ray diffracto- metry, phase equilibrium	illustrates behavior of container materials; dissociates at 1020°C in air to Ir+O ₂ ; see also O ₂ Ru	C. L. McDaniel S. J. Schneider	C. L. McDaniel and S. J. Schneider, "Phase Relations of the Ru-Ir-O ₂ System", J. Res. NBS, 72A, 213 (1968).
$KNaO_4S$ $KNaSO_4$	melt equimolar $K_2SO_4+Na_2SO_4$, annealed over- night at 600°C	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 50.
$K_2Mg_2O_{12}S_3$ $K_2Mg_2(SO_4)_3$	melt K_2SO_4 + $MgSO_4$ at 1000°C	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 40.
$K_2Mn_2O_{12}S_3$ $K_2Mn_2(SO_4)_3$	melt K_2SO_4 + $MgSO_4$, annealed at 500°C for 15 hr.	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 43.
$K_2Na_4O_{12}S_3$ $K_{.67}Na_{1.33}(SO_4)_3$	melt stoi- chiometric amts. K_2SO_4 + Na_2SO_4	x-ray diffracto- metry, petrographic microscopy		H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 48.

$K_2Ni_2O_{12}S_3$ $K_2Ni_2O_{12}S_3$	$K_2SO_4 + NiSO_4$ heated at 750°C, cooled slowly, ground, annealed at 550°C for 30 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 46.
$K_2O_4S_3Zn_2$ $K_2Zn_2(SO_4)_3$	$K_2SO_4 + ZnSO_4$ melted, ground, remelted	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 54.
$K_3NaO_8S_2$ $K_3Na(SO_4)_2$	glaserite stoichiometric K_2SO_4 + Na_2SO_4 melted, annealed at 700°C for 72 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 52.
$LiNaO_4S$ $LiNaSO_4$	equimolar Li_2SO_4 + Na_2SO_4 melted, annealed at 500°C overnight	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 24.
$LiNbO_3$	obtained from CIBA, Rare Metals Div., Summit, N.J.; re-crystallized by W. S. Brower, pulled from melt, annealed under O_2 at 1100°C for 10 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 22.
Li_2O_4S Li_2SO_4	$Li_2SO_4 \cdot H_2O$ heated at 600°C for 24 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $C_2Na_2O_4$, p. 26.

LuO ₄ V LuVO ₄	soln. Na ₃ VO ₄ + Lu ₂ (SO ₄) ₃ + ppt., annealed at 1300°C for 15 hr.	x-ray diffractometry, petrographic microscopy	P. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 37.
Na ₂ NiO ₈ S ₂ ·4H ₂ O Na ₂ Ni(SO ₄) ₂ ·4H ₂ O	crystallized from aq. soln. of Na ₂ SO ₄ + NiSO ₄ at room temp.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 68.
Na ₂ O ₈ S ₂ Zn·4H ₂ O Na ₂ Zn(SO ₄) ₂ ·4H ₂ O	crystallized from soln. of Na ₂ SO ₄ + ZnSO ₄ at room temp.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under C ₂ Na ₂ O ₄ , p. 72.
NbO ₄ Sb SbNbO ₄	solid state rxn. of Sb ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube at 1000-1100°C	see BiNbO ₄	R. S. Roth J. L. Waring	See ref. under BiNbO ₄ .
"NbO ₅ P" "NbPO ₅ " 22Nb ₂ O ₅ ·P ₂ O ₅	solid state rxn. of Nb ₂ O ₅ +P ₂ O ₅ in sealed Pt. tube	x-ray powder diffractometry	E. M. Levin	E. M. Levin and R. S. Roth, "The System Niobium Pentoxide-Phosphorus Pentoxide", J. Solid State Chemistry 2, (1970).
Nb ₂₄ O ₆₂ Ti TiNb ₂₄ O ₆₂	solid state rxn. of TiO ₂ + Nb ₂ O ₅ at 1450°C	x-ray powder and single crystal diffractometry; crystal structure detm.	systematics of "non-stoichiometry"	R. S. Roth and A. D. Wadsley, "Mixed Oxides of Titanium and Niobium: The Crystal Structure of TiNb ₂₄ O ₆₂ (TiO ₂ ·12Nb ₂ O ₅)", Acta Cryst., 18, 724 (1965).

O_2Ru RuO_2	powder	oxidation of Ru by heat treatment in air	x-ray diffractometry, phase equilibrium	illustrates behavior of container materials, dissociates at 1045°C in air to Ru + O_2 ; see also IrO_2	C. L. McDaniel S. J. Schneider	See ref. under IrO_2 .
$(1-x)O_2Th + xCaO$ $0 < x < 0.10$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	spectroscopic analysis	character of point defect; electrical and mechanical relaxation	J. Wachtman, Jr.	J. B. Wachtman, Jr., "Mechanical and Electrical Relaxation in ThO_2 Containing CaO ", Phys. Rev., <u>131</u> , 517 (1963).
$(1-x)O_2Zr + xCaO$ $.04 < x < .20$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	porosity, grain size	spans range of stability of cubic solid solution and includes 2-phase region below $x = .10$ as test of change in character of point defects	J. Wachtman, Jr.	J. B. Wachtman, Jr. and W. C. Corwin, "Internal Friction in ZrO_2 Containing CaO ", J. Res. NBS, <u>69A</u> , 457 (1965).
O_4PrV $PrVO_4$		$Pr_2(C_2O_4)_3 + V_2O_5$ heated at 850°C for 30 min.	x-ray diffractometry, petrographic microscopy		E. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 40.
O_4SbTa $SbTaO_4$		solid state rxn. of $Sb_2O_3 + Ta_2O_5$ in sealed Pt tube at 1000-1200°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.
O_4SbV $SbVO_4$		solid state rxn. of $Sb_2O_3 + V_2O_5$ in sealed Pt tube at 700-865°C	see $BiNbO_4$		R. S. Roth J. L. Waring	See ref. under $BiNbO_4$.

O ₄ SmV SmVO ₄	Sm ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ heated at 850°C for 45 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 47.
O ₄ TbV TbVO ₄	Tb ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ heated at 1400°C for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 56.
O ₄ TmV TmVO ₄	Tm ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ heated at 1400°C for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 57.
O ₄ YbV YbVO ₄	Yb ₂ (C ₂ O ₄) ₃ + V ₂ O ₅ heated at 1400°C for 1 hr.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 58.
Pt	high-purity rod, 1/8 in. dia. x 1.5 in.	Langmuir detm. of vapor pressure as function of temp.; detm. of heat of sublimation	R. C. Paule	See ref. under Ag.
	special prepn. for Office of Standard Reference Materials, proposed SRM # 747			
	SRM # 747 SRM # 680	vapor pressure Langmuir method	E. R. Plante	See above ref.
Re	rod, .25 cm. dia. x 1.5 cm.	vapor pressure	E. R. Plante	E. R. Plante and R. Szwarc, "Vapor Pressure and Heat of Sublimation of Rhenium", J. Res. NBS, 70A, 175 (1966).

$V_2O_5 \cdot 4Y_2O_3$ $4Y_2O_3 \cdot V_2O_5$ $V_2O_5 \cdot 5Y_2O_3$ $5Y_2O_3 \cdot V_2O_5$	$\sim 50\mu$	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	phosphors	E. M. Levin	E. M. Levin, "System $Y_2O_3-V_2O_5$ ", J. Am. Ceram. Soc., <u>50</u> , 381 (1967).
W	high- purity rod, 1/4 in. dia. x 2-1/2 in.	special prep'n. for Office of Standard Reference Materials, proposed SRM # 749	see Pt	see Pt	R. C. Paule	See ref. under Pt.
	polycry- stalline rod	commercial	vapor pressure, Langmuir method	vapor pressure standard; container for high-temp. studies	E. R. Plante	R. Szwarc, E. R. Plante, and J. J. Diamond, "Vapor Pres- sure and Heat of Sublimation of Tungsten", J. Res. NBS, 69A, 417 (1965).

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INFORMATION

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	REFERENCES (Author, Title, Journal)
Al ₂ O ₃ -WO ₃ entire system	polycrystal- line, 1-25 μ	solid state rxn. of com- ponents in sealed Pt tubes	phase equilibria; x-ray powder and single crystal diffraction studies	phase equilibria	J. L. Waring, "Phase Equilibria in the System Aluminum Oxide- Tungsten Oxide", J. Am. Ceram. Soc., 48, 493 (1965). D. C. Craig and N. C. Stephenson, "A Structural Study in the System Al ₂ O ₃ - WO ₃ ", Acta. Cryst., B24, 1250 (1969).
As ₂ O ₅ -9Nb ₂ O ₅ partial system	polycrystal- line, 1-25 μ	solid state rxn. in sealed Pt tube	phase equilibria; x-ray diffraction powder and single crystal structure	illustrates nature of apparent iso- structural phases in related systems; see also: As ₂ O ₅ -9Ta ₂ O ₅ , GeO ₂ -9Nb ₂ O ₅ , 9Nb ₂ O ₅ -P ₂ O ₅ , 9Nb ₂ O ₅ -V ₂ O ₅ , 2Nb ₂ O ₅ -Ta ₂ O ₅ , P ₂ O ₅ -9Ta ₂ O ₅ , 9Ta ₂ O ₅ -V ₂ O ₅	J. L. Waring and R. S. Roth, "Tetragonal Phases of the General Type 10M ₂ O ₅ ·9OM ₂ O ₅ apparently isostruct- tural with Ta ₂ O ₅ . Nb ₂ O ₅ ", Acta Cryst., 17, 455 (1964). R. S. Roth and A. D. Wadsley, "The Crystal Structure of PNB ₉ O ₂₅ (P ₂ O ₅ ·9Nb ₂ O ₅)", Acta Cryst., 18, 643 (1965).
As ₂ O ₅ -9Ta ₂ O ₅	partial system, see comments and ref. under As ₂ O ₅ -9Nb ₂ O ₅				
BO ₃ M MBO ₃	solid solns. of rare-earth borates, M = Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Sm, Tm, Y, Yb; polycrystal- line	solid state rxns. of mix- tures of M ₂ O ₃ + B ₂ O ₃ in sealed Pt tubes	x-ray powder diffractometry; high-temp. x-ray phase equilibria	most specimens exhibit polymorphic inversions	R. S. Roth, J. L. Waring, and E. M. Levin, "Polymorphism of AB ₃ -Type Rare Earth Borate Solid Solutions", Rare Earth Research II, Gordon & Breach, N.Y., (1964), p. 153.

BaFeO _{3-n}	0 > n < 0.5; system in the vicinity of 1:1 cation ratio; polycrystalline, 1-25 μ	solid state rxn. in Au tubes under O ₂	phase equilibria; x-ray diffractometry	illustrates influence of stoichiometry on structure	T. Negas R. S. Roth	T. Negas and R. S. Roth, "Synthesis of Barium Ferrates in Oxygen", J. Res. NBS, 73A, (1969).
BaO-Nb ₂ O ₅	entire system	polycrystalline	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Phase Equilibrium Relations in the Binary System Barium Oxide-Niobium Pentoxide", J. Res. NBS, 65A, 337 (1961).
BaO-Pt	entire system	powders	x-ray diffractometry, phase equilibria	all intermediate phases dissociate; illustrates behavior of container material	S. J. Schneider C. L. McDaniel	S. J. Schneider and C. L. McDaniel, "The BaO-Pt System in Air", J. Am. Ceram. Soc., 52, 518 (1969).
BaSi ₂ O ₅ -Ba ₂ Si ₃ O ₈	entire system	polycrystalline	x-ray powder diffractometry; phase equilibria; single crystal x-ray diffractometry		R. S. Roth E. M. Levin	R. S. Roth and E. M. Levin, "Phase Equilibria in the System Barium Disilicate-Dibarium Trisilicate", J. Res. NBS, 62, 193 (1959).
						R. S. Roth and E. M. Levin, "Polymorphism in Barium Disilicate", Am. Mineral., 44, 452 (1959).
Bi ₂ O ₃ -Nb ₂ O ₅	entire system	polycrystalline	x-ray powder diffractometry; phase equilibria		R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Phase Equilibrium Relations in the Binary System Bismuth Sesquioxide-Niobium Pentoxide", J. Res. NBS, 66A, 451 (1962).

Cr ₂ O ₃ -IrO ₂ entire system	powders	solid state rxns. in Pt, Vycor, Ir containers, conducted in air	x-ray diffractometry; phase equilibria	illustrates behavior of container materials	C. L. McDaniel S. J. Schneider "Phase Relations between Cr ₂ O ₃ and IrO ₂ in Air", J. Am. Ceram. Soc., <u>49</u> , 285 (1966).	C. L. McDaniel S. J. Schneider "Phase Relations between Cr ₂ O ₃ and IrO ₂ in Air", J. Am. Ceram. Soc., <u>49</u> , 285 (1966).
"CrO _n "-SrO "chromium oxide" - SrO entire system	polycrystalline, 1-25 μ	solid state rxns. in air and O ₂	x-ray diffractometry; gravimetric anal.; phase equilibria	illustrates redox behavior of Cr in system	T. Negas R. S. Roth SrO-"chromium oxide" in Air and Oxygen", J. Res. NBS, <u>73A</u> (1969)	T. Negas and R. S. Roth, "The System SrO-"chromium oxide" in Air and Oxygen", J. Res. NBS, <u>73A</u> (1969)
Dy ₂ O ₃ -TiO ₂	partial system, see comments and ref.	under Gd ₂ O ₃ -TiO ₂				
Er ₂ O ₃ -2TiO ₂	partial system, see comments and ref.	under Gd ₂ O ₃ -TiO ₂				
Eu ₂ O ₃ -TiO ₂ Eu ₂ O ₃ -2TiO ₂	partial system, see comments and ref.	under Gd ₂ O ₃ -TiO ₂				
Gd ₂ O ₃ -TiO ₂ entire system	polycrystalline, 1-25 μ	solid state rxn. in unsealed Pt tubes	x-ray diffractometry; polarizing microscopy; phase equilibria	illustrates nature of phases form and relationship to analogous systems; see also: Dy ₂ O ₃ -TiO ₂ , Er ₂ O ₃ -2TiO ₂ , Eu ₂ O ₃ -TiO ₂ , Eu ₂ O ₃ -2TiO ₂ , Ho ₂ O ₃ -2TiO ₂ , Lu ₂ O ₃ -2TiO ₂ , Sm ₂ O ₃ -TiO ₂ , 2TiO ₂ -Tm ₂ O ₃	J. L. Waring J. Schneider Equilibrium Relationships in the System Gd ₂ O ₃ -TiO ₂ ", J. Res. NBS, <u>69A</u> , 255 (1965).	J. L. Waring and S. J. Schneider, "Phase Equilibrium Relationships in the System Gd ₂ O ₃ -TiO ₂ ", J. Res. NBS, <u>69A</u> , 255 (1965).
GeO ₂ -9Nb ₂ O ₅	partial system, see comments and refs.	under As ₂ O ₅ -9Nb ₂ O ₅ ; also see GeO ₂ -9Nb ₂ O ₅ in SECTION V				
Ho ₂ O ₃ -2TiO ₂	partial system, see comments and ref.	under Gd ₂ O ₃ -TiO ₂				

IrO ₂ -SnO ₂ entire system IrO ₂ -TiO ₂ entire system	powders	solid state rxns. in Pt, Vycor, and Ir containers, conducted in air	x-ray diffractometry, phase equilibria	illustrates behavior of container materials	C. L. McDaniel S. J. Schneider "Phase Relations in the Systems TiO ₂ -IrO ₂ and SnO ₂ -IrO ₂ in Air", J. Res. NBS, <u>71A</u> , 119 (1967).
Ln ₂ O ₃ -PdO entire system	powders	solid state rxns. in Pt, Vycor containers	x-ray diffractometry; phase equilibria	all inter-mediate phases dissociate; illustrates behavior of container materials	C. L. McDaniel S. J. Schneider "Phase Relations between Palladium Oxide and the Rare-Earth Sesquioxides in Air", J. Res. NBS, <u>72A</u> , 27 (1968).
Lu ₂ O ₃ -2TiO ₂	partial system, see comments and ref. under Gd ₂ O ₃ -TiO ₂				
9Nb ₂ O ₅ -P ₂ O ₅	partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅ ; also see "Nb ₂ O ₅ P" in SECTION V				
2Nb ₂ O ₅ -Ta ₂ O ₅	partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅				
Nb ₂ O ₅ -V ₂ O ₅ entire system	polycrystalline, 1-25 μ	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; phase equilibria	illustrates nature of multi-phase formation and non-stoichiometric phases; see also 9Nb ₂ O ₅ -V ₂ O ₅	J. L. Waring and R. S. Roth, "Phase Equilibria in the System Vanadium Oxide-Niobium Oxide", J. Res. NBS, <u>69A</u> , 119 (1964).
9Nb ₂ O ₅ -V ₂ O ₅	partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅ ; also see Nb ₂ O ₅ -V ₂ O ₅				
P ₂ O ₅ -9Ta ₂ O ₅	partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅				
Sm ₂ O ₃ -TiO ₂	partial system, see comments and ref. under Gd ₂ O ₃ -TiO ₂				

J. L. Waring and
R. S. Roth, "Effects
of Oxide Additions on
the Polymorphism of
Tantalum Pentoxide
(System Ta_2O_5 - TiO_2)",
J. Res. NBS, 72A, 175
(1967).

illustrates J. L. Waring
various stable R. S. Roth
and metastable
phases formed
in the system

x-ray powder
diffractometry;
high-temp. x-ray
powder diffracto-
metry; phase
equilibria

solid state
rxns. in
sealed Pt
tubes

polycrystal-
line

Ta_2O_5 - TiO_2
entire system

$9Ta_2O_5$ - V_2O_5 partial system, see comments and refs. under As_2O_5 - $9Nb_2O_5$

$2TiO_2$ - Tm_2O_3 partial system, see comments and ref. under Gd_2O_3 - TiO_2

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